Since it is recognized that moisture content does not affect the pliability of hulls to the same degree that it affects the pliability of meats, it is reasonable that the fine meat flakes, where only about 65% of the flake bed (meats portions) was made soft, had a lower bulk density with a corresponding increase in percolation rate over its companion whole meats flake of equal moisture level.

Extractibility. Table III also shows the effect of moisture content upon extractibility. There is little difference between flakes of various moisture contents in the residual oil left in the meal after the 25-minute percolation test. In the case of whole meat flakes the highest extractibility (lowest residual oil) appears to be at a moisture content of approximately 9-10%. In the case of fine meat flakes the extractibility appears to be unaffected by the moisture content. For combined meat flakes there is a trend downward which indicates that an increase in moisture level gives an increased extractibility. However the changes in extractibility are all small; and the results therefore not conclusive.

Fines. Table III shows that moisture content does have a pronounced effect on the fines-producing tendencies of flakes. The fines present before disintegration, which is the case more closely resembling the basket extractor having least flake handling, is at a minimum at a moisture level of approximately 9 to 10%. Decreased moisture causes a large rise in fines so that at approximately 4% moisture the amount of fines is about 12 times greater than at 9% moisture. Increased moisture causes a small rise in fines. At approximately 12% moisture the amount of fines is about 1½ times greater than at 9% moisture.

The fines present after disintegration, which is the case more closely resembling the extractor which agitates the flake bed, is also a minimum at approximately 9-10% moisture. However the variations between the amount of fines at the minimum and the amounts at other moisture levels are not so great as the variations before disintegration.

Conclusions

The results indicate that 9 to 10% moisture in the meats is the best level for the preparation of cottonseed into flakes for solvent extraction. At lower moisture the flakes contain more fines before and after agitation in solvent, and the percolation rate of solvent through a flake bed is lower because of reduced particle size. At higher moisture the amount of oil in the hulls from the beater is greater. Also the flakes produce more fines, and the percolation rate becomes slower because of the softness and pliability of the flaked meat. It is likely that in commercial continuous operations this packing or balling up tendency of the flaked meats may lead to periodic channeling or plugging in the extractor, dryer, conveyors, and filters.

These conclusions have been applied in the continuous pilot plant solvent extraction of three lots of prime cottonseed. The results substantiate the findings herein and will be reported in a separate publication.

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Solvent Extraction IV. The Effect of Temperature on Extraction Rate

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SINCE temperature is a significant factor influencing the rate of extraction of crude lipids from oil seeds with solvents, a knowledge of the relationship between extraction rate and temperature is important in solvent extraction plant design and operation. The literature contains very little quantitative data although several references to the qualitative effect of temperature have been made (1, 3, 5, 6, 8). Karnofsky presented data on the extraction of cottonseed in his Short Course lectures (7). Boucher *et al.* extracted degummed soybean oil from porous clay plates with chlorinated hydrocarbon solvents at several temperatures (2).

In considering the effect of temperature on extraction rate, the experimenter is immediately confronted with the fact that the total "oil" extractable from seeds is itself a variable which may change with the extraction temperature (4, 7, 11). For this reason the results would have little theoretical value unless the residual oil at any time were determined by an analytical method employing the same solvent and temperature as the rate experiment. Of more practical interest is the residual oil as determined by the standard method Ba 3-38 of the A.O.C.S. (10).

For convenience in our laboratory, analyses for residual oil are made by extracting the ground sample with hexane for two hours at the boiling point, which is 150°F. The residuals determined in this way are slightly higher than those found by the standard method.

Experimental Procedure

The Percolation Method (11), modified where necessary, was used in determining extraction rates. The modifications were necessitated by operating conditions other than those specified in the original Percolation Method, namely, the use of solvents other

TABLE I

Extraction Rate Data

Temperature °F.	Residual Oil Content (Per Cent—Dry Basis)					
Series 1. Cottonseed Flakes, Heptane Extracted						
Extn. time	20 min.	30 min.	45 min.	60 min.	90 min.	120 min.
71 (22.0°C.)	6.21	5.34	4.63	4.18	3.57	3.15
192 (88.9°C.)	3.04	2.26	1.70	1.36	0.96	0.69
Series 2. Cottonseed Flakes, Hexane Extracted						
Extn. time	20 min.	30 min.	45 min.	60 min.	90 min.	120 min.
70 (21.1°C.)	5.61	5.02	4.42	4.00	3.43	3.06
100 (37.8°C.)	4.77	4.12	3.54	3.16	2.63	2.26
150 (66.0°C.)	4.00	3.37	2.71	2.29	1.89	1.64
Series 3. Cottonseed Flakes, Isopropanol Extracted (CBM-Water)						
Extn. time	20 min.	40 min.	60 min.	70 min.	120 min.	240 min.
80 (26.7°C.)	10.25	5.57	......	3.75	......	......
123 (50.6°C.)	5.10	......	1.42	......	0.80	......
177 (80.6°C.)	2.61	1.31	0.72	......	0.42	0.34
Series 4. Soybean Flakes, Hexane Extracted						
Extn. time	5 min.	6 min.	10 min.	20 min.	40 min.	60 min.
75 (23.9°C.)	4.08	......	......	1.45	1.13	0.96
95 (35.0°C.)	......	2.82	......	1.31	0.91	......
150 (66.0°C.)	2.06	......	1.20	0.75	......	......
Series 5. Soybean Flakes, Trichlorethylene Extracted						
Extn. time	5 min.	10 min.	15 min.	30 min.	40 min.	60 min.
72 (22.2°C.)	5.68	......	1.85	1.10	0.65	0.54
129 (53.4°C.)	3.53	......	0.95	0.57	......	......
177 (80.6°C.)	1.65	0.67	......	0.30	......	0.21
Series 6. Soybean Flakes, Hexane Extracted						
Extn. time	5 min.	10 min.	20. min.	30 min.	40 min.	80 min.
70 (21.1°C.)[a]	......	......	1.39	......	1.01	0.71
100 (37.8°C.)	......	1.37	0.93	0.64	0.70	......
150 (66.0°C.)	2.09	0.96	0.54	......	......	......
Series 7. Soybean Grits, Heptane Extracted						
Extn. time	10 min.	15 min.	20 min.	30 min.	60 min.	90 min.
84 (28.9°C.)	......	1.42	......	1.13	0.79	0.45
114 (45.6°C.)	......	0.90	......	0.58	0.16	......
192 (88.9°C.)	0.53	......	0.25	0.11	......	......
Series 8. Soybean Grits, Hexane Extracted						
Extn. time	10 min.	15 min.	30 min.	60 min.	90 min.	120 min.
68 (20.0°C.)	......	1.64	1.42	1.13	0.96	0.79
110 (43.3°C.)	......	1.12	0.83	0.57	0.41	0.26
150 (66.0°C.)	0.91	0.74	0.50	0.34	......	......
Series 9. Flaxseed Grits, Heptane Extracted						
Extn. time	10 min.	30 min.	60 min.	90 min.	120 min.	185 min.
70 (21.1°C.)[b]	......	3.48	......	2.60	......	2.00
150 (66.0°C.)	......	1.79	1.13	......	0.60	......
192 (88.9°C.)	2.10	1.40	0.90	......	0.55	......
Series 10. Flaxseed Grits, Hexane Extracted						
Extn. time	10 min.	25 min.	60 min.	90 min.	120 min.	180 min.
70 (21.1°C.)[c]	......	3.31	......	......	1.96	......
100 (37.8°C.)[d]	......	......	......	1.48	......	0.89
125 (51.7°C.)	2.65	1.97	1.33	......	0.93	......
150 (66.0°C.)	2.80	2.09	1.18	......	0.69	......

[a] 70°F.—0.61% in 110 minutes.
[b] 70°F.—0.98% in 740 minutes.
[c] 70°F.—1.22% in 410 minutes, 0.70% in 1,140 minutes.
[d] 100°F.—0.72% in 240 minutes.

than commercial hexane and temperatures other than the boiling point. A series of individual 8-gram samples, taken from a single batch, were run in the percolation apparatus (11) for the desired time intervals, desolventized in air, and the residual oil contents determined by the hexane analysis mentioned above. At temperatures below the boiling point of the solvent, the extracting solvent was supplied to the Butt-tube from a constant-head reservoir, through a heat exchanger and a flow control valve, at the specified rate of flow. Temperature measurements were made with a thermocouple inserted into the bottom of the glass extraction thimble. Moisture contents were determined by vacuum oven drying and toluene distillation, Methods 27.3 and 27.4 of the Association of Official Agricultural Chemists (9).

All factors, other than extraction temperature, which might possibly affect extraction rate were maintained constant in each series of experiments. As nearly as possible, particle size, moisture content, solvent, and solvent rate were kept identical for each rate determination in a series. The modified Percolation Method is not only more laborious and time-consuming than the original but also gives less reproducible results on materials which do not lend themselves readily to uniform sampling in small quantities so that more points are required to establish a reliable rate curve.

Relation of Temperature and Extraction Rate

Table I presents the extraction rate data for soybeans, cottonseed, and flaxseed grouped in series. In each series, samples derived from the same batch were extracted with the same solvent. Table II gives descriptions of the batches and the solvents used for each series. In Figure 1 the data of Series 4 for the

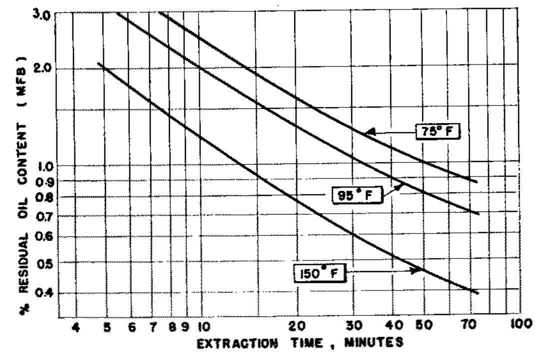

FIG. 1. Rate of extraction of soybean flakes (Series 4) at three temperatures.

extraction of soybean flakes with commercial hexane at three temperatures are plotted to show the effect of temperature on the extraction rate curves obtained. The data of Table I were all plotted in the manner illustrated in Figure 1. From the resulting curve families, cross plots were made showing the relation of temperature and "Time to 1%" (5, 10), defined as the extraction time in minutes required to reduce the oil seed to a dry-basis oil content of 1.0%. This is a criterion of extraction rate. Figure 2 shows the log-log cross plot for each series with curves drawn precisely through the times to 1% read from the extraction rate curves.

Attempts were made, without success, to correlate the extraction rate data with changes in viscosity of the solvent which might reasonably be expected to influence the rate of diffusion of the solvent into, and the solvent-oil solution out of, the porous structure of the oil seed. Boucher (2) showed that the extraction of degummed soybean oil from clay plates with solvent followed the laws governing simple molecular diffusion. He was able to correlate extraction rates for this system with the product of the viscosities of the solvent and the oil. However it has been shown that simple molecular diffusion does not explain the results obtained in the extraction of crude soybean oil from soybean flakes (4, 7). Boucher's correlation did not give a relationship between temperature and extraction rate for the data of this paper more consistent than did any other empirical plot. Moreover

TABLE II

Series Descriptions

Series No.	Material	Preparation	Wt. % moisture	Extracting solvent
1	Cottonseed	Commercial plant flaking rolls	11.6	Heptane
2	Cottonseed	Commercial plant flaking rolls	11.6	Hexane
3	Cottonseed	Commercial plant flaking rolls	11.8	CBM isopropanol-water (88% by wt. isopropanol)
4	Soybeans	Commercial plant flaking rolls	7.6	Hexane
5	Soybeans	Commercial plant flaking rolls	8.0	Trichlorethylene
6	Soybeans	Commercial plant flaking rolls	8.4	Hexane
7	Soybeans	Ground in laboratory attrition mill	7.0	Heptane
8	Soybeans	Ground in laboratory attrition mill	7.0	Hexane
9	Flaxseed	Ground in commercial 5-high rolls	9.2	Heptane
10	Flaxseed	Ground in commercial 5-high rolls	9.2	Hexane

the large change of extraction rate with temperature compared to Boucher's data for diffusion is indicative of a different extraction mechanism for actual oil seeds.

In the literature, explanations for the effect of temperature on extraction are based on the viscosity of the solvent and the solubility of some of the components of the crude oil (3, 8). The total effect probably results from the combination of the two with each predominating in different temperature ranges.

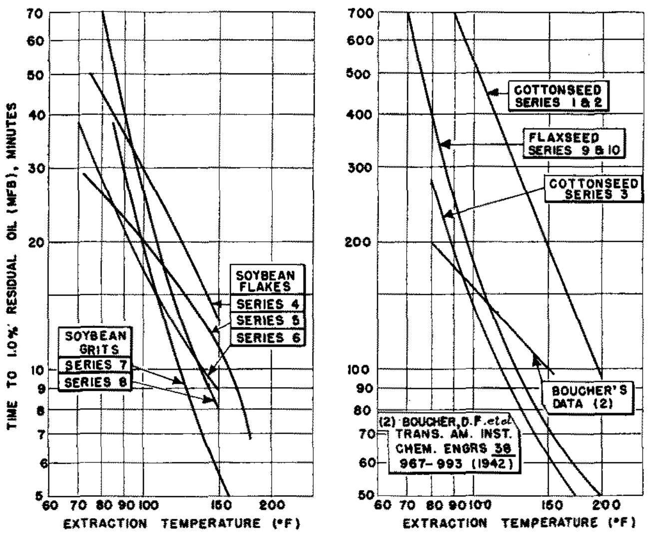

FIG. 2. Effect of temperature on extraction rate.

Application of the Data

Within the range of temperatures of practical interest in commercial extraction (from 100°F. to the boiling point of the solvent), straight lines can replace the curves of Figure 2. The extraction rates near room temperature demonstrate the inadvisability of operating at lower extraction temperatures. In Figure 3 such straight lines are drawn for the materials of commercial importance. Maximum deviation of the straight lines from the curves of Figure 2 is 10%, but the original data probably do not justify much greater precision. The general equation of these straight lines is:

$$\text{Log } \theta = n \log t + \log K$$

$$\text{or, } \theta = K t^n$$

where, θ is the time to 1%, t is the temperature in degrees Fahrenheit, K is the ordinate intercept at the 1°F. line, and n is the slope of the line (negative). Since the time to 1% varies with the nth power of the temperature, the value of n is a convenient measure of the variation of extraction rate with temperature. The values of n for the lines shown in Figure 3 are: Series 1, cottonseed flakes extracted with heptane, —2.4; Series 2, cottonseed flakes extracted with hexane, —2.4; Series 3, cottonseed flakes extracted with CBM isopropanol-water, —1.9; Series 4, soybean flakes extracted with hexane, —2.0; Series 5, soybean flakes extracted with trichlorethylene, —2.0; Series 6, soybean flakes extracted with hexane, —1.6; Series 9, flaxseed grits extracted with heptane, —1.9; Series 10, flaxseed grits extracted with hexane, —1.9. Materials of little practical significance, not shown in Figure 3, gave slopes of —2.5 for soybean grits extracted with hexane and heptane and —1.5 for the clay plate data of Boucher. As an approximation the time to 1% in minutes varies inversely with the square of the extraction temperature in degrees Fahrenheit in the practical temperature range.

A hypothetical plant situation will serve to illustrate the importance of extraction temperature in plant operation. A basket, or Bollmann, extractor operating on a 60-minute cycle at 110°F. is producing spent flakes with a residual oil content of 1.0%. Taking into consideration drainage time, co-currency, and dumping time, the effective extraction time is 40 minutes. Using the inverse square relationship, the time required to reach the same residual oil content at 140°F. would be about 24 minutes. Accordingly, the retention time in the extractor could be reduced about 25% (cycle time reduced to about 45 minutes) with a corresponding 25% increase in extractor capacity.

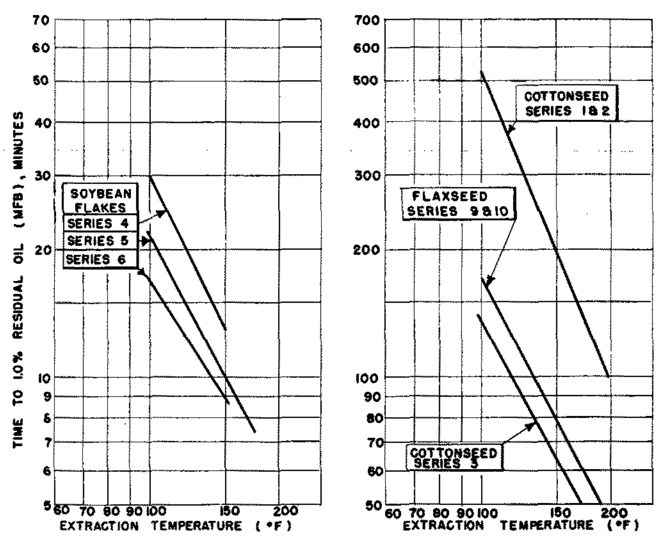

FIG. 3. Effect of temperature on extraction rate.

Summary

The temperature has a considerable effect on the rate of extraction of crude oils from vegetable oil seeds with solvents. Quantitative data have been presented relating extraction rate and temperature for soybeans, cottonseed, and flaxseed extracted with several solvents. These data were obtained by the Percolation Method, modified where necessary.

Since no satisfactory theoretical basis for correlation could be established, the results were correlated empirically. For all practical purposes the time in minutes required to reduce the oil seed to 1.0% residual oil content on a dry basis varied inversely with the square of the extraction temperature in degrees Fahrenheit.

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Oil Production From African Oil Palms in Honduras

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THE search for new crops for Latin America and the world shortage of edible oils led to an investigation, commencing in 1942, of the possibilities of producing palm oil in Honduras. Large areas of land were available where both soil and climatic conditions were considered satisfactory for development of this important crop.

A collection of African oil palm varieties comprising the following selections was available in the Plant Introduction Garden of the United Fruit Company at Lancetilla, Honduras:

Variety	Origin
Reinking No. 7-C-1	Java
Reinking No. 8-C-1	Java
Reinking No. 32-B-3	Java
Reinking No. 19	Federated Malay States
Reinking No. 33	Federated Malay States
Reinking No. 51	Federated Malay States
Fairchild No. 1192	Africa (Diwakkawakka)
Sumatra	Sumatra (U. S. Rubber Co.)

This planting of selected strains from the East Indies, Malay States, and West Africa was made in 1926. In 1942 the stand included 60 palms of Java variety, 137 from Malaya, 43 from Africa, and 200 of Sumatra varieties. Fruit from these palms was observed and compared over a period of two years in order to select the highest variety for propagation.

Processing methods and yields per acre were studied over a period of five years in a pilot plant at La Lima, Honduras, utilizing fruit from a 23-acre farm, established in the years 1936 to 1938 by a local planter with mixed seed.

Varietal Studies

Mature fruit was harvested monthly from individual palms in the Lancetilla collection and their weights were recorded. Due to age and crowded condition of this planting, yield figures are not necessarily indicative of production under more favorable circumstances, but the difference between varieties was quite evident (Table I). The number of fruit bunches harvested annually per palm varied from four to seven. The largest bunch harvested was of the Java variety and weighed 124 lb.

TABLE I

Number and Weight of Fruit Bunches From Mature Palms

Variety	Harvested	Average weight per bunch in lb.
Java	104	45.8
Sumatra	218	45.4
Malay	102	38.8
Africa*	5	24.6

* One bunch only.

Over a period of two years one bunch of mature fruit of each variety was analyzed monthly for yield of palm and kernel oil. The Java variety was the highest producer of both palm and kernel oil per bunch and also gave the highest average field weight per bunch (Table II).

TABLE II

Yield Data of Representative Bunches of Three Palm Varieties (In Pounds)

	Java		Sumatra		Malay	
	Range	Average	Range	Average	Range	Average
Gross weight per bunch	37-96	59	34-87	53	30-93	47
Net weight fruit	28-62	42	28-57	32	21-54	31
Palm oil	5.2-17.7	8.3	3.6-14.9	7.2	2.4-10.5	6.1
Kernel oil	1.0- 3.8	1.9	1.0- 2.8	1.4	.5- 2.6	1.2

These varieties of oil palms were also classified as to the percentage of oil contained in the pericarp and kernel; by the weight of fruit, pericarp, and kernel; and by the thickness of pericarp and shell. Samples from identical fruit used in the yield studies were