Sesame Oil. II. Some Chemical and Physical Properties of the Oils From Different Varieties of Sesame Seed[1]

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SESAME seed is one of the oldest oilseeds known to man. It is mentioned in the ancient Hebrew and Egyptian scripts, such as the Ebers Papyrus. Different varieties and the manifold uses of the oil are described in ancient Sanskrit literature (1). Cultivation of this crop in the past has been restricted mainly to China, India, the Near East, Africa, and some countries in Central and South America. Shattering of the seed and non-uniform maturation make mechanical harvesting a difficult problem consequently cultivation of sesame has hitherto been restricted to regions where there has been an abundant and cheap supply of labor.

Intensive programs for the improvement of sesame are in progress principally in Venezuela and the United States. New varieties are being developed which might eventually be adapted for mechanical harvesting (2). Information concerning the characteristics of the seed oils from known varieties of domestic origin is desirable, especially in view of the results reported in the first publication of this series (3).

The present report is concerned with the results of an investigation of the oils from three different domestic varieties of sesame seed grown at Clemson, S. C., and Lincoln, Neb., and identified as follows: SO-1, Clemson 4522 (1948); SO-2, Clemson 4520 (1948); SO-3, Nebraska 1025-3 (1948). For purposes of comparison, the oil of another batch of Nicaraguan seed variety (SO-4), reported in the first paper of this series (3), was extracted and reinvestigated after the seed had been stored for a period of 14 months.

Experimental

Crude Oil. Data with reference to the extraction of the Nicaraguan seed were similar to those presented in a previous paper (3) in this series. The seeds of the Clemson and Nebraskan varieties were flaked to an average thickness of 0.010 inch and immediately charged into a batch extraction unit (4). Hexane at 100°F. was pumped through a charge of 100 pounds of flaked seed at an average rate of 35 gallons per hour for 15 hours. The miscella was concentrated at a temperature of 180°F. under partial vacuum in a current of carbon dioxide. The meal was first air-dried for 12 hours and then oven-dried at 120°F. for 6 hours. Analyses of the original seed and extracted meals are given in Table I.

The physical and chemical characteristics of the four crude oils were determined by the Official Methods of the American Oil Chemists' Society (5), except for the unsaponifiable matter (6), thiocyanogen value (7), and hydroxyl number (8), for which the methods described in the references were used. The physical and chemical characteristics of the four crude oils are given in Table II. The Nicaraguan variety (SO-4) had a slightly higher iodine and lower thiocyanogen value as well as a substantially higher content of unsaponifiable matter and a higher optical rotation.

TABLE I

Composition of Sesame Seed and Extracted Meals

Constituent	SO-1	SO-2	SO-3	SO-4
Seed				
Moisture, %	4.82	4.98	5.33	4.90
Oil, %	54.8	55.6	51.2	49.5
Nitrogen, %	3.11	3.37	4.08	4.20
Protein (N × 6.25), %	19.4	21.1	25.5	26.4
Extracted meal				
Moisture, %	8.04	5.50	5.34	6.09
Oil, %	2.34	15.40[a]	3.47	6.47
Nitrogen, %	7.10	6.74	8.68	.....
Protein (N × 6.25), %	44.4	41.8	54.2	.....

[a] Incomplete extraction because of channeling in the extractor.

TABLE II

Characteristics of Crude Sesame Oils

Characteristics	SO-1	SO-2	SO-3	SO-4
Color, Lovibond Y/R, 5¼ in. cell	70/2.4	70/16.7	70/4.3	70/4.0
Specific gravity, 25°/25°	0.9188	0.9192	0.9193	0.9207
Optical activity, $\alpha_D^{25°}$, 1 dm. cell	0.93	0.95	1.02	1.44
Titer, °C	23.1	23.7	23.6	23.5
Cold test[a]	Does not pass test	Does not pass test	Passes test	Passes test
Volatile matter, %	0	0.03	0	0.02
Free fatty acids, (as oleic), %	0.5	1.8	1.5	2.0
Hydroxyl number	1.1	4.1	3.3	4.2
Iodine value, Wijs	111.5	111.6	109.8	112.8
Thiocyanogen value	76.2	76.1	75.3	75.0
Saponification value	188.2	188.3	188.0	186.9
Unsaponifiable matter, %	1.63	1.72	1.78	2.28
Reichert-Meissl value	0.2	0.2	0.1	0.1
Polenske value	0.4	0.5	0.2	0.1
Refractive index n^{40}	1.4660	1.4671	1.4658	1.4664

[a] Test carried out with the refined and bleached oils.

In Table III is given the fatty acid composition, calculated on the basis of results of the determination of iodine and thiocyanogen values, saturated acids by the Bertram oxidation method as modified by Pelikan and Von Mikusch (9), except that sintered glass filter sticks were used for the filtration of the magnesium soaps, and the spectrophotometric absorption after alkali-isomerization (10).

The different methods used for the determination of the fatty acid composition gave values which, in general, agreed with one another. The percentages of saturated fatty acids calculated from iodine-thiocyanogen values agreed especially well with those obtained by the modified Bertram oxidation method.

Refined Oil. The crude sesame oils were refined according to the official method (5) of the American Oil Chemists' Society for solvent-extracted soybean oils, using a 0.1% excess of 12° Bé. lye. The oils were bleached with 5.5% of official American Oil Chemists' Society earth and also with 2% of "B. C. clay." The results of the refining and bleaching tests are given in Table IV, from which it may be seen that the refining losses were uniformly low and practi-

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TABLE III

Fatty Acid Composition of the Crude Sesame Oils

Oil	Glycerides of						
	Linoleic %		Oleic %		Saturated %		
	A[a]	B[b]	A[a]	B[b]	A[a]	B[b]	C[c]
SO-1	43.4	41.4	42.1	45.4	12.8	11.2	13.4
SO-2	43.8	46.1	41.6	36.4	12.9	15.5	13.4
SO-3	42.4	43.6	42.1	39.1	13.6	15.1	13.8
SO-4	46.6	46.6	37.3	36.1	13.8	14.4	13.8

[a] Calculated from iodine-thiocyanogen values, corrected for unsaponifiable matter.
[b] Spectrophotometric analysis after alkali-isomerization, corrected for unsaponifiable matter.
[c] Modified Bertram oxidation method.

cally identical bleach colors were obtained with the "B. C. clay" as with the official clay.

About 20 pounds of each of the crude sesame oils were refined in a stainless steel laboratory refining kettle, using a 0.1% excess of 12° Bé. lye with agitation for 45 minutes at room temperature and 20 minutes at 60°C. The soap was allowed to settle overnight, the refined oil drawn off, filtered, and bleached without drying. Bleaching was carried out in a stainless steel, open bleaching kettle by stirring for 5 minutes with 2% "B. C. clay" at 120°C. The bleached oils were immediately filtered with the aid of suction on a filter precoated with a small quantity of filter-aid. The colors of the filtered oils were identical with those obtained by the official bleaching test.

TABLE IV

Data on Refining and Bleaching Tests

Characteristic	SO-1	SO-2	SO-3	SO-4
Free fatty acids, (as oleic), %	0.5	1.8	1.5	2.0
Refining loss, %	1.4	4.5	4.4	4.7
Color refined oil, Lovibond Y/R	35/1.2	35/1.6	35/0.6	20/1.2
Color bleached oil, 5.5% AOCS earth	1/0.5	1/0.5	2/0.4	3/0.6

Hydrogenation. The refined and bleached oils were selectively hydrogenated at a temperature of 350°F. and 15 p.s.i. pressure with 0.1% of dry-reduced, electrolytically precipitated nickel hydroxide (11) as catalyst. In order to follow the course of the hydrogenation, about 12 pounds each of oils of SO-1 and SO-2 were hydrogenated under the above-mentioned conditions in the batch hydrogenator (12). One-pound samples were withdrawn at predetermined intervals corresponding to definite decreases in the iodine value of the oils. The oils were filtered and the fatty acid composition, the refractive index, consistency, and stability were determined on each sample. The consistencies of the hydrogenated oils were determined at 25°C. by the micropenetration technique by Feuge and Bailey (13) with samples chilled for 16 hours and tempered for 30 minutes at 25°C. prior to testing. Isooleic acid was determined by the Twitchell lead salt-alcohol method of the American Oil Chemists' Society (5). Data for the composition and characteristics of the hydrogenated oils are given, Table V.

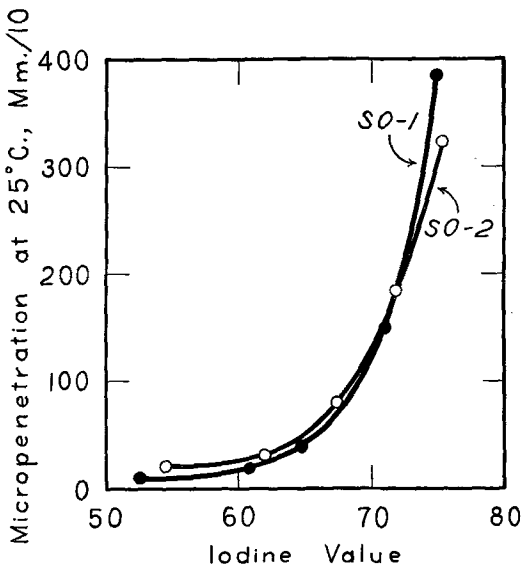

FIG. 1. Variation of consistency with iodine value for two progressively hydrogenated sesame oils.

It will be noted that the stability of the oils increases rapidly during hydrogenation and reaches remarkably high values. This marked stability of hydrogenated sesame oil has been studied and reported on in detail in another article of this series (14).

TABLE V

Composition and Characteristics of Hydrogenated Sesame Oils

Sample No.	Iodine value Wijs	Thiocyanogen value	Glycerides of								Refractive index, n_D^{60}	Micropenetration at 25°C., 0.1 mm.	Stability A.O.M. hrs.[d]
			Linoleic, %		Oleic, %			Saturated, %					
					Total		Iso-[c]						
			A[a]	B[b]	A[a]	B[b]		A[a]	B[b]	C[c]			
SO-1-0	111.5	76.2	43.4	41.4	42.1	45.4	.....	12.8	11.2	.....	1.4587	.....	19.5
-1	88.0	75.5	15.1	16.6	72.0	68.2	9.2	11.4	13.3	12.9	1.4558	.....	96
-2	79.6	74.0	6.5	6.2	79.6	79.7	.....	12.4	12.4	.....	1.4549	.....	182
-3	74.9	73.6	1.1	1.9	84.9	83.0	15.8	12.5	13.4	13.5	1.4543	385	325
-4	70.7	70.5	0	0.1	82.7	81.6	.....	15.8	16.6	.....	1.4538	151	605
-5	64.8	64.8	0	0	76.3	75.0	24.5	22.2	23.3	23.8	1.4532	41	740
-6	60.7	60.5	0	0	70.9	70.2	.....	27.6	28.1	.....	1.4526	20	825
-7	52.8	52.8	0	0	62.2	61.0	29.0	36.3	37.3	37.9	1.4516	10	1040
SO-2-0	111.6	76.1	43.8	46.1	41.6	36.4	.....	12.9	15.5	.....	1.4588	.....	17.7
-1	88.3	74.3	16.9	15.7	68.6	70.2	9.3	13.0	12.1	14.3	1.4557	.....	63
-2	80.0	73.3	7.8	7.2	77.2	77.8	.....	13.5	13.1	.....	1.4549	.....	181
-3	75.2	72.3	3.1	2.9	81.2	81.1	17.4	14.2	14.2	17.4	1.4542	209	330
-4	71.3	69.6	1.6	1.1	79.6	80.1	.....	17.3	17.0	.....	1.4536	185	470
-5	67.0	67.2	0	0.3	79.4	76.8	21.9	19.1	21.1	23.9	1.4533	77	583
-6	62.1	61.6	0	0	71.8	71.6	.....	26.7	26.6	.....	1.4530	31	740
-7	57.7	57.5	0	0	67.4	66.5	23.9	31.1	31.7	33.3	1.4525	20	910

[a] Calculated from iodine-thiocyanogen values, corrected for unsaponifiable matter.
[b] Spectrophotometric analysis after alkali-isomerization, corrected for unsaponifiable matter.
[c] Twitchell lead-salt-alcohol method.
[d] Time necessary to reach peroxide number of 100 m.e./kg. at 97.7°C.

TABLE VI

Composition and Characteristics of Sesame Oils Hydrogenated to Shortening Consistency

No.	Iodine value Wijs	Thiocyanogen value	Glycerides of									Refractive index, n_D^{60}	Micropenetration at 25°C., 0.1 mm.
			Linoleic, %		Oleic, %			Saturated, %					
					Total		Iso-						
			A[a]	B[b]	A[a]	B[b]		A[a]	B[b]	C[c]	D[d]		
SO-1	70.0	68.3	1.7	0	78.0	81.1	37.0	18.8	17.2	18.1	19.0	1.4528	53
SO-2	69.4	68.4	0.8	0	79.2	80.3	36.2	18.6	18.0	17.8	19.3	1.4531	51
SO-3	68.4	67.5	0.6	0	78.3	79.0	37.2	19.6	19.2	18.4	20.0	1.4532	45
SO-4	69.7	67.7	1.9	0	77.1	80.3	37.4	18.5	17.8	18.1	19.1	1.4540	51

[a] Calculated from iodine-thiocyanogen values, corrected for unsaponifiable matter.
[b] Spectrophotometric analysis after alkali-isomerization, corrected for unsaponifiable matter.
[c] Modified Bertram oxidation method.
[d] Twitchell lead-salt-alcohol method.

Data for the variations in the micropenetrometer values (consistency) as a function of iodine values is shown graphically in Figure 1 from which it may be determined that hydrogenation of sesame oil to an iodine value of approximately 69 produces a fat in the range of shortening consistency. The refractive index as a function of iodine value is shown in Figure 2. An approximately linear relationship is obtained.

Small portions (200 g. each) of oils SO-1 to SO-4, refined and bleached as previously described, were hydrogenated under conditions approximately similar to those described above to an approximate iodine value of 69.0. The hardened oils were steam-deodorized in a laboratory-model all-glass deodorizer.

Samples of 150 grams each of the four hydrogenated oils were deodorized for 2 hours at 200°C. and 1-mm. pressure with a total throughput of stripping steam of approximately 60 grams per sample. The resultant oils were completely free of odor but darkened slightly during deodorization. For comparison, the corresponding refined and bleached, unhydrogenated oils were also deodorized in exactly the same manner. After deodorization the samples were evaluated in the same manner as the progressively hydrogenated oils with the results shown in Table VI.

Summary

Four varieties of sesame seed, grown in South Carolina, Nebraska, and Nicaragua, were solvent extracted in a pilot-plant and the oils examined with respect to their physical and chemical characteristics. Only slight variations were found in these constants and in the composition of the oils.

The oils were refined, bleached, hydrogenated to shortening consistency, and deodorized. The refining losses were low (1.4% to 4.7%) and the bleach colors light (1-3 yellow/0.4-0.6 red, Lovibond scale). Changes in composition and in values of the refractive index and plasticity during hydrogenation were similar to those reported for other oils of similar characteristics. The hydrogenated oils reached a shortening consistency at an iodine value of about 69. A.O.M. stability values for the hydrogenated oils were high.

The data presented indicate that conventional methods of processing sesame oil can be applied with good results. Changes in constants and fatty acid composition during hydrogenation are essentially similar to those occurring in other vegetable oils, such as cottonseed or peanut oils.

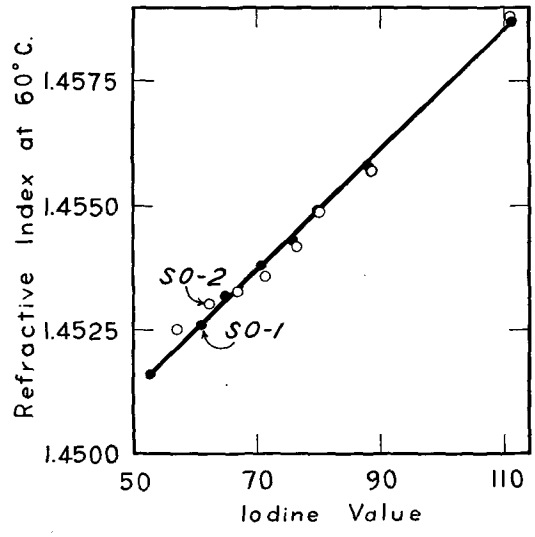

FIG. 2. Variation of refractive index with iodine value for two progressively hydrogenated sesame oils, ● SO-1, ○ SO-2.

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REFERENCES

1. Hedebrand, A., Landw. Vers.-Sta., *51*, 45-81 (1898).
2. Langham, D. G., and Rodriguez, M., El Ajonjoli (Sesamum Indicum L.) su cultivo, explotacion y mejoramiento, Ministerio de Agricultura y Cria, Boletin, No. 2, 1945, Caracas, Venezuela.
3. Andraos, V., Swift, C. E., and Dollear, F. G., Sesame Oil. I. Properties of a Solvent-Extracted Sesame Oil, J. Am. Oil Chem. Soc. *27*, 31-34 (1950).
4. Pominski, J., Molaison, L. J., Crovetto, A. J., Westbrook, R. D., D'Aquin, E. L., and Guilbeau, W. F., Oil Mill Gazetteer, *51* (12), 33-39 (1947).
5. American Oil Chemists' Society, Official and Tentative Methods, 2nd ed., Chicago, 1946.
6. Cocks, L. V., Report of the Subcommittee on Determination of Unsaponifiable Matter in Oils and Fats and of Unsaponified Fats in Soaps, Analyst, *58*, 203-211 (1933).
7. Lambou, M. G., and Dollear, F. G., Oil & Soap, *22*, 226-232 (1945).
8. West, E. S., Hoagland, C. L., and Curtis, G. H., J. Biol. Chem., *104*, 627-634 (1934).
9. Pelikan, K. A., and Von Mikusch, J. D., Oil and Soap, *15*, 149-150 (1938).
10. American Oil Chemists' Society, Report of the Spectroscopy Committee, J. Am. Oil Chem. Soc., *26*, 399-404 (1949).
11. Bailey, A. E., Industrial Oil and Fat Products, Interscience, New York, 1945, pp. 591-593.
12. Bailey, A. E., Feuge, R. O., and Smith, B. A., Oil & Soap, *19*, 169-176 (1942).
13. Feuge, R. O., and Bailey, A. E., Oil & Soap, *21*, 78-84 (1944).
14. Budowski, P. Menezes, F. G. T., and Dollear, F. G., Sesame Oil. V. The Stability of Sesame Oil, J. Am. Oil Chem. Soc. (In press.)

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