

American Safflower Seed Oil

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SAFFLOWER seed oil is obtained from the seed of *Carthamus tinctorius* L., a plant which has been extensively cultivated for many years in India, Egypt, and Turkestan as an oilseed crop. The seed, which somewhat resembles small sunflower seed, contains from 20 to 30 per cent of oil, and the kernels contain from 46 to 50 per cent. The oil is produced in considerable quantities in India, where it is used as a drying oil in the paint and varnish industries. It possesses value also as an edible oil and for use in the manufacture of soap.

With a view to introducing this crop in the United States as a source of oil to supplement the domestic production of drying oils, the Bureau of Plant Industry, U. S. Department of Agriculture, has for several years made experimental plantings of safflower in various localities of the Northwest and in the middle western States under the direction of Mr. Frank Rabak.*

Much information with regard to the agronomic requirements of the crop has been obtained, and also technological information regarding the production and handling of the oil. Crushing tests on a semi-commercial scale have been made with American-grown seed for the production of the oil and press cake. This oil has been tested in the laboratory and by manufacturers of paints and varnishes and has been found to possess value as a drying oil in these industries. The press cake has been found useful as a stock feed. Hot-pressed oil from seed grown in Montana has been investigated in this laboratory and its approximate composition determined.

The chemical and physical characteristics are given in Table 1. The iodine number of this oil, from 138 to 145, is somewhat higher than that usually reported for the foreign oil. The percentages of saturated and unsaturated acids were determined by the lead-salt-ether method, and corrections were made for the small quantity of unsaturated acids that are precipitated and weighed with the saturated acid fraction (*Jour. Amer. Chem. Soc.* 1920, 42, 2398; *Cotton Oil Press*, 1922, 6, 41). The

percentage of unsaturated acids has also been corrected for the unsaponifiable matter that remains with the unsaturated fraction.

TABLE I
SAFFLOWER OIL

Specific gravity 25/25°9243
Refractive index at 25°	1.4744
Acid value	5.56
Iodine number (Hanus)	149.3
Iodine number (Wijs)	149.1
Saponification value	190.5
Acetyl value	12.5
Reichert-Meißl value	0.2
Polenske number	0.1
Hexabromide %	0.4
Unsaponifiable matter %59
Saturated acids (corrected) %	5.93
Unsaturated acids (corrected) %	87.72
Iodine No. of Unsat. Acids	156.0

Unsaturated Acids

THE linolenic acid was separated and weighed as hexabromide according to the Washburn and Steele procedure (*Ind. Eng. Chem.* 1920, 12, 521). The average of 3 determinations in close agreement was 0.40 per cent of hexabromide, which is equivalent to 0.14 per cent of linolenic acid in the original oil. Having determined the percentage of linolenic acid, it is possible to calculate the percentages of oleic and linolic acid from the iodine number of the unsaturated fraction, 156.0, and the theoretical iodine numbers of the three acids; 274.1 for linolenic acid, 181.4 for linolic acid, and 90.1 for oleic acid. In this way the percentage composition of the unsaturated acids tabulated below was calculated.

	Unsat. Acid Fraction Per Cent	Unsat. Acids in oil Per Cent	Glycerides of Unsat. Acids Per Cent
Linolenic Acid	.16	0.14	.15
Linolic Acid	71.82	63.00	65.8
Oleic Acid	28.02	24.58	25.7
	100.00	87.72	91.65

Saturated Acids

THE saturated acids which were separated from the mixed fatty acids of the oil, by the lead-salt-ether method, were esterified with absolute methyl alcohol, using dry hydrogen chloride gas (*Jour. Amer. Chem. Soc.* 1920, 42, 1200), and the resulting esters were fractionally distilled under diminished pressure. The data for the distillation are given in Table 2. The preliminary distillation was made from a 500 cc. Claisson flask, giving 5 fractions and a residue of undistilled esters which were redistilled in the order indicated in the table,

*Safflower, an oilseed crop, is adapted to Northern Great Plains. Separate from Yearbook of Agriculture, 1927. No. 1013.

from a 150 cc. Ladenburg fractionation flask. Seven fractions and a small residue were obtained.

TABLE II

FRACTIONAL DISTILLATION OF METHYL ESTERS OF SATURATED ACIDS

(123.6 g. taken for distillation)

(Preliminary distillation under 4 mm. pressure)		Fraction	Temperature °C	Wt. Gms.
		A	176-9	21.50
		B	180-3	22.05
		C	184	21.55
		D	185-8	22.20
		E	190-200	21.90
		Residue		14.80
(Final distillation under 2 mm. pressure)				
	Fractions A + B added...	1	163-8	9.20
		2	170.3	21.70
	Fraction C added.....	3	174-6	23.10
	Fraction D added.....	4	176-8	22.70
	Fraction E added.....	5	180-3	22.59
	Residue added.....	6	185-195	14.30
		7	200-220	9.45
	Residue			.75

The iodine numbers and the saponification values of these seven fractions were determined and are recorded in Table 3. The small final residue was saponified with alcoholic potash, and the fatty acids were liberated with hydrochloric acid, collected, dried, and crystallized from 12 cc. of absolute alcohol. The crystalline fatty acid (0.620 g.), which melted at about 77°, was recrystallized; then it gave a melting point of 80-80.5°. Equal quantities of this acid and lignoceric acid of known purity were intimately mixed, and the melting point of the mixture was 80 to 80.5°. Examination of alcoholic mother liquors failed to give evidence of any other saturated acids. The iodine numbers of the various fractions are the measure of the contaminating unsaturated acids, and from these values the percentages of the esters of these unsaturated acids were calculated.

fraction 1 (column 5, Table 3) is between methyl myristate (242.3) and methyl palmitate (270.3), and indicates that this fraction contained both esters, whereas the molecular weights of fractions 2, 3, 4, 5 and 6 suggest that they contain various proportions of methyl palmitate and stearate (298.4), but that fraction 7 is a mixture of methyl arachidate (326.4) and lignocerate (382.5).

In order to test the correctness of these deductions, the free fatty acids were recovered from several of the fractions by saponifying them with alcoholic potash, removing the alcohol, dissolving the soaps in water, and decomposing them with hydrochloric acid. The constituent saturated acids entirely freed from mineral acid were isolated by fractional crystallization from ethyl alcohol. Their identity was established by the melting points and by observing whether or not they were lowered when the substances were mixed with equal quantities of the respective acids which they were suspected of being, the purity of which had been established by analysis.

The deductions drawn from the molecular weights of the saturated acids were confirmed as follows:

Lignoceric acid $C_{24}H_{48}O_2$ melting at 80-80.5° was isolated from residue; arachidic acid $C_{20}H_{40}O_2$, melting at 77° was obtained from fraction 7; stearic acid $C_{18}H_{36}O_2$ melting at 68-69° was separated from fractions 5 and 6; palmitic acid $C_{16}H_{32}O_2$ melting at 63° was isolated from fractions 1 and 2. Ten crops of crystals were obtained from the fractional crystallization of the fatty acids from fraction 1 by gradually reducing the volume of the alcoholic solution and finally adding small quantities of water. The small tenth crop melted at

TABLE III
SAFFLOWER OIL
RESULTS OF ANALYSES OF FRACTIONS OBTAINED BY
DISTILLING THE METHYL ESTERS

Frac- tion	Iod. No.	Sap. Val.	Esters of Unsat. Acids %	Molec. Wt. of Esters of Sat. Acid	Myristic Acid		Palmitic Acid		Stearic Acid		Arachidic Acid		Lignoceric Acid	
					%	Gram	%	Gram	%	Gram	%	Gram	%	Gram
1	2.5	209.3	1.68	267.6	8.92	.8213	84.25	7.7510						
2	2.7	205.9	1.79	272.1			87.11	18.9013	6.05	1.3127				
3	2.6	204.9	1.72	273.5			82.57	19.0748	10.67	2.4088				
4	2.9	203.2	1.96	275.7			78.40	17.7975	14.62	3.3204				
5	3.5	198.8	2.28	282.0			54.07	12.2151	38.76	8.7571				
6	5.3	188.4	3.42	298.0			0.13	.1860	91.68	13.1100				
7	11.2	171.4	7.53	330.3							82.72	7.8170	6.15	.5812
Residue														.6200
Totals							.8213	75.9257	28.9090	7.8170				1.2012

From these percentages and the saponification values the mean molecular weight of the saturated acid esters in the several fractions were calculated. The mean molecular weights indicate what saturated acids may be present in each fraction. For example, the mean molecular weight of the saturated acid esters in

54-5°, and it was found to be myristic acid $C_{14}H_{28}O_2$.

The quantities of saturated acids in the fractions were calculated from the mean molecular weight of their esters and the theoretical molecular weights of the two esters in each fraction, the results of which are given in Table 3.

TABLE IV
SAFFLOWER OIL
SATURATED ACIDS

Acids	Acids in saturated acid fraction		Acids in original oil	Glycerides in original oil
	Grams	Per cent	Per cent	Per cent
Myristic	0.82	0.71	.04	.04
Palmitic	75.93	66.21	3.93	4.12
Stearic	28.91	25.21	1.49	1.56
Arachidic	7.82	6.82	.41	.42
Lignoceric	1.20	1.05	.06	.06
	114.68	100.00	5.93	

In Table 4 the percentage composition of the saturated acids is given in column 2. These values have been calculated to the basis of the original oil and are given in column 3. Column 4 gives the equivalent percentages of the glycerides.

Summary

THE chemical and physical characteristics of a sample of hot pressed oil from safflower seed grown in Montana have been determined. This oil was found to contain 87.72 per cent of unsaturated acids, and 5.92 per cent of saturated acids.

The composition of the oil has been determined with the following results, and, for comparison, results for sunflower seed and soy

bean oils previously obtained, are also given.

Glycerides of:	Safflower Oil	Sunflower Oil ¹	Soy Bean Oil ²
	Per cent	Per cent	Per cent
Oleic acid	25.7	33.4	33.4
Linolic acid	65.8	57.5	51.5
Linolenic acid15	0.0	2.3
Myristic acid04	0.0	0.0
Palmitic acid	4.1	3.5	6.8
Stearic acid	1.6	2.9	4.4
Arachidic acid4	.6	.7
Lignoceric acid06	.4	.1
Unsaponifiable Matter59	1.2	.6

¹ G. S. Jamieson and W. F. Baughman, J. Am. Chem. Soc. 1922,44,2952. This oil gave an iodine number (Hanus) of 130.8.

² W. F. Baughman and G. S. Jamieson, *ibid* 2948. This oil gave an iodine number of 128.

It will be observed that safflower oil contains a considerably larger proportion of linolic acid and less oleic acid than either of the other two oils, and this fact would account for its superior drying power.

J. T. Baker Issues New Stock

An offering of 10,000 shares of the common stock of the J. T. Baker Chemical Company, Phillipsburg, N. J., at \$12.50 a share was reported to be over-subscribed. The Baker company was organized in 1904 as a New Jersey corporation and has been chiefly engaged in the manufacture and sale of chemicals used for analytical purposes in industrial and educational laboratories. It also manufactures a large variety of chemicals for pharmaceutical and industrial uses. The company has recently purchased the business of the Dissosway Chemical Company, Brooklyn, and that of the Taylor Chemical Corporation, Cascade Mills, N. Y. Its factory property at Phillipsburg comprises 10 acres.

By action of the Board of Governors of the New York Stock Exchange the 110 par common stock of Spencer Kellogg & Sons, linseed crushers and vegetable oil refiners, has been listed for trading on the Exchange floor.

Vegetable Oils Duties

Vegetable oils imported into Syria must now be certified by a French consul.

The export surtax on olive oil originating in the second, fourth and fifth regions of Tunis has increased from 2 francs to 2.50 francs per 100 net kilos, by a recent decree.

A proposal has been made for replacing the existing surtax of 7 percent ad valorem on foreign olive oil and soya bean oil imported into French West Africa (except Ivory Coast and Dahomey) by the following specific rates, according to the Journal Officiel, Paris:—Olive oil, 105 francs per 100 net kilos; soya bean oil, pure or mixed in any proportion with any other edible oil, 157.50 francs per 100 net kilos.

The plant of the Southern Cotton Oil Company, at Ft. Gaines, Ga., is reported to be under water, in the flood that has been causing much trouble in that territory recently.