acid from several of the possible structures or could arise from minute amounts of residual eleostearate.

All our evidence goes to show that a cyclohexadiene of type I is the probable primary product of cyclization. Under the influence of heat some double bonds migrate, giving rise to structures II–VI. Structure VII is probably the final result of the double bond migration. It is surmised that VII also contains a ring because the cyclic monomer does not form a urea complex.

Summary

Evidence is presented for the presence of various structural types in the cyclic product obtained by thermal polymerization of methyl β-eleostearate under mild conditions. Of these types a 1,2-dialkyl-3,5-cyclohexadiene is probably the primary product.

Acknowledgment

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Seed Germination as an Index of Potential Free Fatty Acid Content of Sesame Oil[1]

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INTRODUCTION of the new indehiscent (non-shattering) varieties, Rio (1) and Palmetto (5), of sesame may make possible economic production of this ancient crop as a domestic source of edible oil and protein supplement. In the harvest of normal dehiscent or shattering type of sesame, hand labor is required to prevent excessive loss of seed. While the new indehiscent varieties may be machine-harvested, very careful adjustment of the combine used for threshing is required to prevent excessive damage to the seed. The seed capsules of indehiscent sesame require considerable force for threshing, and the seeds are tender and easily damaged. Even microscopic cracks in the seed coat cause the seed to lose viability. Free fatty acid content of oil in dead seeds apparently increases much more rapidly than in live seeds. Hoffpauir, Petty, and Guthrie (4) reported that free fatty acid content of oil from dead cottonseed greatly exceeded that of viable seed. Beroza and Kinman (3) found little change in free fatty acid in seed which had been hand-threshed to prevent damage to the seed after storage at 100°F. for five to 11 months.

This report presents the results of experiments designed to estimate the relationship between viability of seed at harvest time and the free fatty acid in oil from seed which had been stored for various periods after threshing.

Experimental Materials and Methods

Twenty-three samples of seed of the variety Rio were obtained from a machine-harvesting experiment designed to obtain information on requirements for maximum threshing efficiency and minimum seed damage. These samples were combined from the windrow during the period from October 14 through October 20, 1954. Rainfall on the windrows, totaling 2.61 in. between September 27 and October 7, 1954, caused only slight damage, as indicated by practically the same viability in seed combined before and after the rains. Seed samples were cleaned immediately after threshing and stored in paper envelopes at room temperature. On October 23 germination tests of these samples were started. Two lots of 100 seed were taken from each sample, treated with Spergon to prevent mold, placed between sheets of moist filter paper in Petri dishes and incubated at 28°C. for eight days. In counting the seed, any portion of over one-half seed was considered as a whole seed. Seeds which showed any indication of germination were considered viable. Non-viability shown in these tests was used as a measure of threshing damage.

Free fatty acid content of the oil (hereinafter referred to as FFA) was determined by a method derived from methods of the A.O.A.C. (2) and A.O.C.S. (6). A 10-g. sample of seed was ground in petroleum ether with a Sargent centrifugal wet mill. The extracted oil was filtered with suction into a tared flask, the ether evaporated, and the oil weighed. Oil content of these samples was approximately 49%. FFA was determined and reported as percentage of oleic acid in the oil.

A random sample of plants taken from the same field were hand-threshed on December 10 to avoid seed damage. The hand-threshed seed lots were used as check samples in all later runs for FFA and viability.

Experimental Results

Hand-threshed seed (a total of 180 samples representing two replications of each of 90 indehiscent sesame strains grown in yield tests in 1954) averaged 98.3% germination while machine-threshed seed from the same plots averaged 67.1% germination. Loss of viability was apparently almost entirely caused by damage in threshing.

Germination of seed of 10 varieties produced in 1953 ranged from 98.7 to 17.3% three months after harvest; FFA ranged from 0.22 to 3.18% five months after harvest.[2] The correlation coefficient computed between the data for viability and FFA was —.926

[1] Presented at annual meeting, American Oil Chemists' Society, Houston, Tex., April 23–25, 1956.

TABLE I

Viable Seed and Free Fatty Acid Content of Oil from Sesame Seed as Affected by Threshing Treatments and Time in Storage

Sample No.	Viable seed, percentage		Free fatty acids (as oleic), percentage						
	After		After						
	6 days	170 days	32 days	59 days	87 days	115 days	143 days	171 days	522 days
34 (hand threshed check)	97.0	98.0	......	.22	.25	.26	.39	.40	.52
24	82.5	63.0	.73	......	......	......	......	2.02	4.80
25	80.5	66.0	.70	......	......	......	......	1.97	4.18
30	79.0	66.0	.74	......	......	......	......	2.02	4.02
26	78.0	62.0	.63	1.00	.94	1.32	1.55	1.92	4.04
27	74.0	67.5	.65	.91	1.38	1.14	1.20	1.57	3.38
33	74.0	72.5	.79	......	......	......	......	2.08	4.24
29	71.5	70.0	.87	......	......	......	......	2.03	4.21
23	71.0	60.5	.53	......	......	......	......	1.86	4.31
32	70.5	68.5	.84	......	......	......	......	1.86	4.20
28	68.0	63.0	.73	.96	1.19	1.22	1.50	1.88	3.68
19	67.0	58.0	1.01	......	......	......	......	2.92	5.89
22	65.5	67.5	.73	......	......	......	......	2.14	4.74
21	63.5	51.0	.87	1.29	1.72	1.82	2.21	2.47	5.41
31	60.0	61.5	.89	......	......	......	......	2.53	4.92
20	60.0	59.0	.76	......	......	......	......	2.29	5.00
13	60.0	61.0	.90	1.43	1.81	1.91	2.19	2.84	5.58
18	59.0	57.5	.79	......	......	......	......	2.48	5.28
12	54.0	48.5	.95	1.47	1.84	2.02	2.45	2.75	5.48
16	53.5	48.5	1.17	......	......	......	......	3.41	6.61
17	53.5	48.0	.77	......	......	......	......	2.37	5.10
14	51.0	39.5	1.19	......	......	......	......	3.65	3.65
11	47.0	46.0	.96	......	......	......	......	3.39	6.08
15	46.0	41.0	1.11	1.73	2.21	2.34	2.94	3.69	6.30
Mean[a]	64.7	58.6	.84	1.26	1.58	1.68	2.01	2.44	4.96

[a] Mean does not include the hand threshed check.

with 8 degrees of freedom, indicating a rather close relationship between these two variables.

Viability and FFA data for 24 samples of seed produced in 1954 are shown in Table I. Viability and FFA were determined on seven of these samples at 28-day intervals for the first 170 days of the storage period. Viability data are shown for only the first and last dates at which germination tests were made. The results from the analysis of these data indicated that values obtained at intermediate dates did not differ significantly from those for the first date. Highly significant decreases in viability after 170 days indicate factors other than threshing damage were beginning to affect viability.

FFA data for oil from seed which had been stored for 32 days after threshing and at 4-week intervals thereafter to 171 days and after 522 days are also presented in Table I. FFA increased rather consistently throughout the period.

Correlation coefficients computed between a) viability at first and last dates, b) viability six days after harvest and FFA at each date, c) viability at 170 days and FFA at 171 days, and d) FFA 32 days after harvest and at all other dates are presented in Table II. The results from these analyses indicated that all of the correlation coefficients were highly significant at the 1% level.

The correlation coefficient (.830) between viability of the seed after six and 170 days indicates that threshing damage is an important factor in determining seed germination percentage in sesame.

Highly significant negative correlation coefficients between viability six days following threshing and FFA at various dates suggest that the reduction in germination percentage occurs immediately and remains relatively constant whereas the FFA increases with the length of the storage period. Both the FFA at a given time and the rate at which FFA increases with time are functions of threshing damage; this is shown by calculated regression lines presented in Figure 1.

[2] The assistance of personnel of the Southern Regional Research Laboratory at New Orleans in making the FFA determinations in this preliminary experiment is gratefully acknowledged.

TABLE II

Correlation Coefficients for Viable Seed and Free Fatty Acid Content of Oil in Sesame Seeds for Various Periods of Storage After Harvest

Correlation between	Number of samples	Correlation coefficient[a]
Viable seed after 6 days and viable seed after 170 days	23	.830
Viable seed after 6 days and FFA after 32 days	23	—.707
Viable seed after 6 days and FFA after 32 days	7	—.988
Viable seed after 6 days and FFA after 59 days	7	—.945
Viable seed after 6 days and FFA after 87 days	7	—.939
Viable seed after 6 days and FFA after 115 days	7	—.930
Viable seed after 6 days and FFA after 143 days	7	—.937
Viable seed after 6 days and FFA after 171 days	23	—.832
Viable seed after 6 days and FFA after 171 days	7	—.937
Viable seed after 6 days and FFA after 522 days	23	—.799
Viable seed after 170 days and FFA after 171 days	23	—.844
Viable seed after 170 days and FFA after 171 days	7	—.872
FFA after 32 days and FFA after 59 days	7	.975
FFA after 32 days and FFA after 87 days	7	.950
FFA after 32 days and FFA after 115 days	7	.968
FFA after 32 days and FFA after 143 days	7	.972
FFA after 32 days and FFA after 171 days	23	.902
FFA after 32 days and FFA after 171 days	7	.967
FFA after 32 days and FFA after 522 days	23	.854

[a] All r values exceed the 1% level of probability.

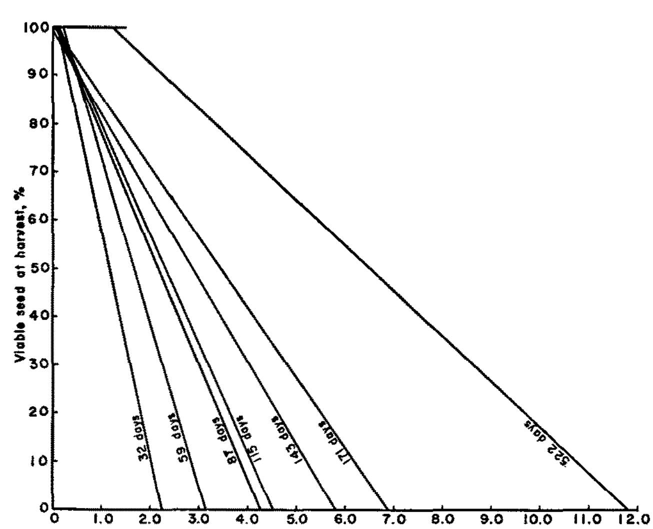

FIG. 1. Relationship between viable seed at harvest and free fatty acid content of oil from sesame seed stored for various lengths of time.

The correlation coefficient between viability after 170 days and FFA after 171 days was only slightly higher than that between viability after six days and FFA at 171 days or 522 days. In this experiment germination tests made immediately after harvest would appear to provide a method for estimating the potential FFA of sesame seed at later dates.

Although correlation coefficients between FFA after 32 days and at later dates were high, it is unlikely that FFA values determined immediately after harvest would be of any predicative value since FFA increased throughout the storage period.

Present data do not provide sufficient evidence upon which to base an accurate prediction of potential FFA of oil from sesame seed under all conditions. A more reliable germination test should be developed since duplicate determinations sometimes showed significant variations in viability. More varieties should be studied to determine whether they all react alike. The effect of different conditions of seed production and storage should be further investigated. If later studies show that the relation between viability at harvest and FFA remains linear, a mathematical formula or a nomograph to estimate FFA at any given date from a knowledge of germination at harvest time may be developed.

Summary

Damage to sesame seed by mechanical threshing equipment resulted in loss of seed viability immediately. FFA content of oil in damaged seed increased gradually after harvest. High correlation coefficients were obtained between viability immediately following harvest and free fatty acid content of oil in sesame seed at various dates after harvest. The results of the analyses of the data indicate that viability of the seed at harvest may be of value in estimating the free fatty acid content of oil for various storage periods.

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The Difficultly Extractable Lipides of Cottonseed Meats, Their Composition and Effect on the Refining Characteristics of the Crude Oils[1]

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THE TREND IN RECENT YEARS to the use of solvent extraction for processing cottonseed has been pronounced. About 21% of the cottonseed produced in 1952–53 were processed by mills using the solvent extraction process in some form, as compared to only 11.5% in 1951–52 (6); and a number of mills have converted to solvent extraction since 1953.

Incentive for the more extensive use of the solvent-extraction processes has been that of obtaining greater yields of crude oil, as about 96–98% of the total lipides can be recovered by the solvent-extraction processes as compared to 83–93% by the mechanical expression methods.

Many mill operators, as well as personnel engaged in cottonseed processing and utilization research, have suggested that solvent extraction for maximum crude oil recovery has been over-emphasized at the expense of product quality and value. This viewpoint appears to be supported by the fact that solvent-extracted meals tend to be dusty and to have poor pelleting characteristics (8) and that many solvent-extraction plant operators have encountered oil-color problems.

Bull and Hopper (2) demonstrated that the lipides extracted from soybeans by successively more exhaustive extractions increased in impurities content with an increasing degree of total lipides extraction. Pons, Hoffpauir, and Thurber (12) have shown that the lipides extracted from cottonseed cake by the solvent-extraction step of the combination screw-press, solvent-extraction process is high in impurities and of lower quality than that obtained by the screw-pressing step. However no systematic study of the composition of the difficultly extractable lipides of cottonseed, or of their effect on the refining characteristics of the crude oils in which they are included, has been reported in the literature. It is the purpose of this paper to report the results of such a study.

In this work cottonseed meats from two different lots of seed were prepared for extraction by three different methods. The prepared meats were extracted with hexane by a series of successive stepwise extractions, and the fractional portions of the total meats-lipides so obtained were quantitatively isolated and analyzed. Crude oils corresponding to various percentages of total lipides extraction were then reconstituted from the crude lipides fractions and evaluated for refining characteristics.

Materials

The cottonseed meats used in this study were from prime delintered 1953 crop seed, hulled as required for use, and were essentially whole meats as nearly free from hulls as could be produced without hand-picking. Two lots of seed, one from the vicinity of Greenwood, Miss., and one from the El Paso, Tex., area were used. The whole meats from the two lots of seed contained, on a moisture-free basis, 35–36% and 39–40% of oil, respectively, and are designated

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[2] One of the laboratories of the Southern Utilization Research Branch, Agricultural Research Service, U. S. Department of Agriculture.