

Flavor Reversion in Soybean Oil.

III. The Preparation and Flavor Characteristics of a Simulated Soybean Oil^{1,2}

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THE various theories (1, 2, 3, 4, 5) that have been advanced to explain the cause of flavor reversion in fats and oils may be classified into three main categories. The source of the objectionable flavors and odors may be considered (a) a glyceride constituent, (b) a non-glyceride constituent, or (c) a complex formed by interaction of these two. From a study of the properties of a simulated linseed oil and from other evidence, Armstrong and McFarlane (1) concluded that the causative agent of flavor reversion in linseed oil was present in the glyceride portion of the oil. In the case of flavor reversion in soybean oil the causative factor has not yet been definitely located among the three possibilities cited. It was thought that one or more of the possible causes might be eliminated by studying the flavor characteristics of a simulated soybean oil whose constituent fatty acids originated from sources other than soybean oil.

The present paper deals with an investigation of the flavor characteristics of a triglyceride mixture whose unsaturated fatty acid composition approximated that of soybean oil. The flavors appearing in the synthetic product under various conditions were compared with those in soybean and cottonseed oils.

Experimental

Preparation of Fatty Acids. Highly purified stearic and palmitic acids were prepared from crude acids (Neofat) by fractional distillation of their methyl esters and subsequent repeated fractional crystallization of the respective acids from ethyl ether. Stearic and palmitic acids were the only saturated acids used in the synthesis of the simulated soybean oil.

Oleic acid (I. V. 89.9) was prepared from the mixed fatty acids of olive oil by fractional distillation of the methyl esters and low-temperature crystallization by the methods of Wheeler and Riemenschneider (6) and Brown and Shinowara (7).

Linoleic acid (I. V. 180.3) was prepared by debromination (8) of tetrabromostearic acid obtained from cottonseed oil fatty acids. After distillation of the crude acid the purified product was stored in a sealed ampule at -30° C.

Linolenic acid (I. V. 265.6) was prepared from linseed oil fatty acids by a modification of the method of Rollett (9). The hexabromostearic acid, recrystallized from toluene (10), was suspended in ether and treated with three times the theoretical amount of zinc dust. The hexabromostearic acid, which was nearly insoluble in the solvent, gradually disappeared during the course of the debromination reaction. The linolenic acid, after distillation, was stored in a sealed ampule at -30° .

Preparation of Simulated Soybean Oil. A mixture of fatty acids containing 103.5 g. of linoleic acid, 20.3 g. of linolenic acid, 39.2 g. of oleic acid, 32.5 g. of

palmitic acid, and 7.5 g. of stearic acid, was esterified with 21.2 g. of glycerol at a temperature of 125° C. according to the procedure of Wheeler, Riemenschneider, and Sando (11). The esterification was followed by measuring the water evolved and in seven hours the reaction was 95% completed.

The product was light yellow and was liquid at room temperature. It possessed a characteristic flavor which was not entirely removed by three successive deodorizations (12). A bland and colorless product was finally obtained by passing a petroleum ether solution of the oil through a column of activated alumina (>90% recovery) and then deodorizing the recovered oil. The fatty acid composition as determined spectrophotometrically (13) and the analytical constants of the simulated oil before and after chromatographic treatment on a column of activated alumina are given in Table I.

TABLE I
Analysis of Simulated Soybean Oil

| | Before Alumina Treatment | After Alumina Treatment |
|--|--------------------------|-------------------------|
| Iodine Value..... | 129.2 | 126.3 |
| Saponification Equivalent..... | 284.9 | 287.6 |
| Refractive Index at 30° | 1.47015 | 1.46993 |
| Fatty Acid Composition | | |
| Linolenic acid..... | 11.2% | 11.8% |
| Linoleic acid..... | 46.2% | 46.8% |
| Oleic acid..... | 23.2% | 18.3% |
| Saturated acids..... | 19.4% | 23.6% |

Accelerated Flavor Stability Tests. (1) **Light Exposure**—Ten-ml. samples of the oils were exposed in Petroff culture flasks under nitrogen to a G-E reflector-drying lamp as previously described (14).

(2) **Heat Treatment**—Fifty-ml. flasks containing 10 ml. of the oil samples were evacuated to less than one mm. pressure and then inserted into an oil bath maintained at 200° C. After one hour, the flasks were removed, cooled, and the contents diluted with an equal quantity of mineral oil. Heating of the oil samples *in vacuo* instead of in air was necessitated by the lack of antioxidants in the simulated oil.

Taste Panel. The flavor testing was done as described earlier (15) by a panel of six well-trained individuals. To obtain a permanent record of the flavor judgments of the panel, the 10-point flavor scoring system of Dutton *et al.* (16) was employed.

Results and Discussion

The results given in Table II show the comparative flavor scores of samples of simulated soybean oil versus soybean oil, cottonseed oil versus soybean oil, and cottonseed oil versus simulated soybean oil after exposure to the G-E reflector-drying lamp and after heat treatment. It will be noted that the flavor ratings obtained in the comparison of the simulated soybean oil with soybean oil are quite close to those for the comparison of cottonseed oil and soybean oil. This might lead one to anticipate little difference

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TABLE II
Flavor Tests of Simulated Soybean, Soybean,
and Cottonseed Oils

| Oil | Light Exposure | | Heat Treatment 1 hr. |
|------------------------|----------------|-------|-------------------------|
| | 0.5 hr. | 1 hr. | |
| Simulated Soybean..... | 8.5 | 5.4 | 7.0 |
| Soybean..... | 5.3 | 3.6 | 3.8 |
| Cottonseed..... | 8.2 | 7.8 | 7.7 |
| Soybean..... | 5.4 | 4.6 | 4.5 |
| Cottonseed..... | 8.2 | 8.2 | 7.5 |
| Simulated Soybean..... | 4.8 | 5.0 | 4.0 |

between the flavor stabilities³ of cottonseed oil and soybean oil. Actually, as shown in Table II, there is a marked difference. Possibly this apparent discrepancy is due to the fact that there is no standard basis of comparison between the various pairs examined, i.e., the second sample of any given pair is judged solely on the basis of the value assigned to the first. To obviate this lack of a standard of reference all possible pairs of the three oils had to be tested. From the information in Table II the oils may be arranged in the following order of decreasing flavor stability: cottonseed > simulated soybean > soybean.

The flavor panel unanimously agreed that the flavors produced by heat and light treatment of the simulated oil were distinctly different from those appearing in soybean oil under the same conditions. The flavors were difficult to describe, but grassy, hay-like, and other flavors typical of reverted soybean oil were absent. Drying and persistent aftertastes evident in the soybean oil were sometimes encountered in the simulated oil.

The effect of tocopherol on the flavor quality and stability of the simulated soybean oil was next investigated. Refined soybean oil is reported (17) to contain 0.02% of α -tocopherol and 0.10% of γ -tocopherol. A sample of the simulated oil containing these concentrations of added α - and γ -tocopherol was exposed to the G-E lamp for one hour and compared to a similar sample of the simulated oil containing no added antioxidant. The flavor scores were 7.5 for the

³A distinction is made in this paper between the terms flavor stability and flavor reversion. The latter is applied only to the characteristic taste and flavor of light and heat-treated soybean oil. The former is used in a broader sense to designate the relative flavor qualities among the several oils examined.

former sample and 5.5 for the latter. Despite this improvement in flavor stability, the tocopherol did not appear to change the quality of the flavor and no reversion effects were apparent. This was also found to be the case when a sample of the simulated oil containing 0.10% of α -tocopherol was shelf-stored in light at room temperature for nine days.

Conclusions from organoleptic observations have to be drawn with considerable caution. They are no more reliable, in the ultimate sense, than are the organs of taste and smell of the individual members of the flavor panel. Thus far the results would tend to indicate that the ordinary fatty acid constituents of soybean oil are not entirely responsible for the flavor characteristics of reverted soybean oil. Likewise, the hypothesis that linolenic acid is the sole causative agent does not appear likely although it is possible that this acid contributes to the flavor instability of soybean oil, particularly to the persistency and drying effects of the reverted oil.

Summary

A simulated soybean oil has been synthesized from purified fatty acids. The flavor characteristics of the oil after heat and light treatment are described and compared to those of soybean and cottonseed oils.

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Report of Seed and Meal Analysis Committee May, 1946

THE Seed and Meal Analysis Committee has under study the methods of analysis of cottonseed, peanuts, tung fruit and their meals, and of soyflour. The Subcommittee for Cottonseed and Cottonseed Meal reports as follows:

In considering the problem of reexamining the methods of the A.O.C.S. for moisture in cottonseed and cottonseed products it was felt that fundamental data on weight losses under varying conditions of type of oven, temperature, time, and sample preparation should be obtained. Through the generous cooperation of the Southern Regional Research Laboratory a thorough program of investigation was undertaken, following the pattern used in "Determination of Moisture in Peanut Kernels," Hoffpauir, *Oil & Soap*, November, 1945. The materials used were whole and crimped cottonseed, fumed ground cottonseed, whole and ground cottonseed meats, and cottonseed meal. As this work by Hoffpauir and Petty has been published in *Oil & Soap*, November, 1946, the results will not be fully reviewed here. It is a valuable addition to the literature on this subject.

The weight loss curves indicated that (1) the drying of whole seed 12-16 hours (overnight) is most reliable and that (2) drying crimped seed 5 hours at 101°C. gave results several tenths per cent lower. (3) Whole seed dried 2½ hours at 130°C. gave results in close agreement with overnight at 101°C. (4) Fumed ground seed showed a small continued weight loss beyond the official drying period of 2 hours at 101°C. (5) Cottonseed meal showed a very small weight loss between two and three hours' drying at 101°C.

The committee undertook collaborative work, under routine conditions, on the above materials at, and with some modifications of, the drying periods named. Within the limits chosen, the variations were in general agreement with the weight loss curves of Hoffpauir and Petty. See Table I.

On the basis of these data the majority of the committee agree that:

1. Drying whole cottonseed 12-16 hours (overnight) at 101°C. is justified as the official method for moisture.
2. The procedure of drying crimped seed 5 hours at 101°C. should be removed from official methods as low results