Key Factors and Recent Advances in the Flavor Stability of Soybean Oil

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Abstract

This review of research at the Northern Regional Research Laboratory covers the effect of metallic impurities, their inactivation, proper protection for the oil, hydrogenation-winterization and other new aspects. Commercial application of this information has shown that soybean oil stability can be improved. These improvements in quality of the soybean oil extend its usefulness. Selective hydrogenation catalysts that may lead to still further improvements have been discovered.

Introduction

SOYBEAN OIL HAS BECOME the major food fat of the United States. During the past decade the factory consumption of soybean oil has approximately doubled from about 2.26 billion pounds in the year ending October 1955 to 4.12 billion pounds in the year ending October 1965 (14). Figure 1 shows these increases by the individual major outlet: margarine, shortening, and salad and cooking oils. Much of our research at Peoria has been centered on improving the quality of salad and cooking oils. It is particularly gratifying to us that the biggest increase in reported edible use has been in this category. An average increase of more than 100 million pounds each year—or more than 1 billion pounds in 10 years—is truly phenomenal. Earlier reviews about our research on the flavor stability of soybean oil have appeared in journals, books and symposium proceedings (5,8,9,15).

These increases in quality and in use—though hand-in-hand—have not come easily. Soybean oil made in the finest of commercial plants may be poor in quality if adequate controls are not exercised. For example, the replacement of one steel valve with a brass valve in a refining plant is said to be able to lower quality of the soybean oil produced. Copper is known to affect the oil adversely when present in trace amounts. Selected soybean oils purchased in 1962 under a specification varied in flavor score from 3.7 to 7.6 initially. These same oils had peroxide values of 72 to 22 after 8 hr under the conditions of the active oxygen method (AOM) (1). The best of these oils would be considered only of average quality now. In 1966 commercial samples examined had initial flavor scores of 7.8 to 8.5 and an 8-hr AOM peroxide value of 1.5.

Early Improvements

Improvement in the United States in the 1940's and early 1950's came through studies of metal impurities and their inactivation. Even today, however, some research workers fail to appreciate the importance of metallic impurities and their proper inactivation in the finished oil. Also, some refiners still do not fully appreciate either the importance of avoiding metallic contamination, or the problems of inactivating traces of metals in the oil. Of course, they may also choose to carry out operations in supposedly economic ways that lead to lower quality. Recent improvements in some U.S. refining plants have made possible the commercial preparation of unhydrogenated soybean oil that equals hydrogenated-winterized soybean oil and other available edible oils in most of the qualities that we usually measure. Further improvement of this unhydrogenated oil appears not only possible but desirable.

Studies on combined techniques of hydrogenation and winterization have led to the nationwide distribution of soybean salad and cooking oils. When properly carried out with an oil of good initial quality, hydrogenation-winterization leads to a more stable edible grade of oil for cooking and for salads. The discovery of selective catalysts for hydrogenating linolenate faster than linoleate with little or no formation of saturated esters suggests that it may be feasible to prepare salad and cooking oils even from oils with higher linolenate content and without winterizing the hydrogenated oil.

Metallic Impurities

Iron and copper have long been known to be detrimental to the flavor stability of vegetable oils (9). Clear-cut information that metallic impurities profoundly affected soybean oil was lacking, however, until we published our first research on the flavor stability of soybean oil (11). We found that small amounts of copper (0.01 to 0.03 ppm) and iron (0.03 ppm) were sufficient to lower the flavor quality and stability of an oil (23). The effects of these metals on samples of good quality soybean oil are shown in Figure 2.

In addition to valves and other metallic surfaces in the refining plant, the soybean itself can be a source of copper and iron. Metallic content of four "native" soybean oils untouched by metal was 0.7 to 0.8 ppm iron and 0.3 to 0.5 ppm copper (19).

Processing the oil through an extraction plant can increase these amounts to more than 5 ppm iron and 0.5 ppm copper (19). Proper alkali refining lowers the content of iron and copper substantially. Studies show that the metallic content of crude oil need not necessarily affect stability of the finished salad oil adversely. Improper removal or inactivation of the metallic impurities does, however, give an inferior oil for salad and cooking purposes. Bleaching and deodorization can increase the amount of metallic impurities too much in plants with supposedly no

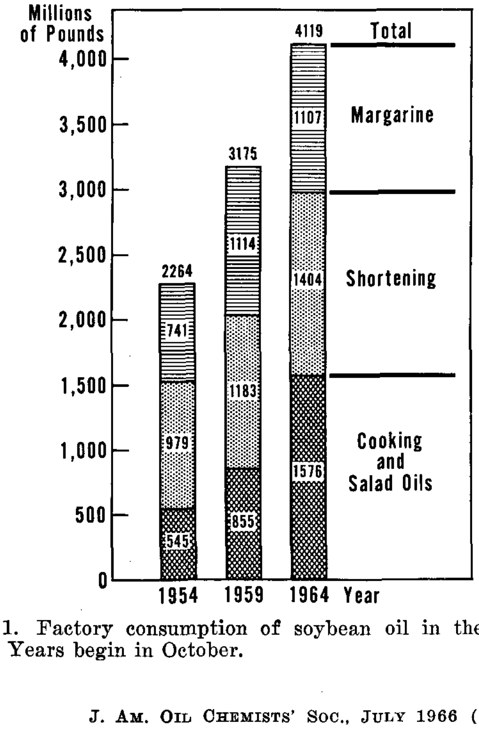

FIG. 1. Factory consumption of soybean oil in the United States. Years begin in October.

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[1] No. Utiliz. Res. Dev. Div., ARS, USDA.

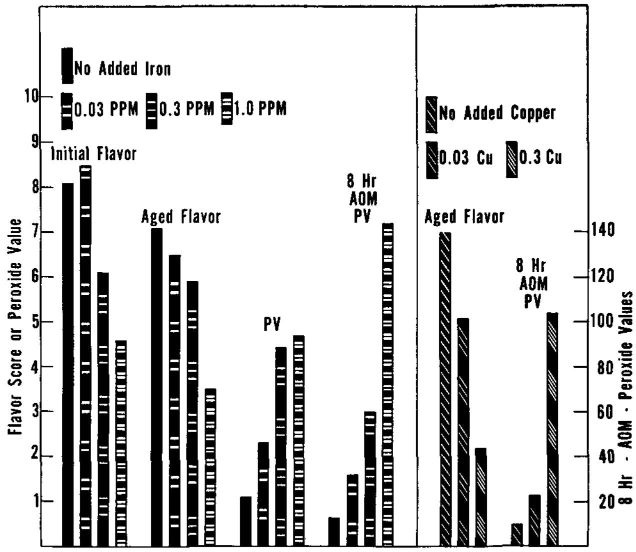

FIG. 2. Effect of added iron and copper on flavor score and oxidative stability of soybean oil.

brass valves but with older steel deodorizers. For example, metallic content can be as low as 0.09 to 0.01 ppm of iron and copper after alkali refining but increase to 1.5 and 0.3 ppm of iron and copper, respectively, in such plants. Ingredients of foods made with oil may also be sources of metallic contamination. The many factors related to trace metals in soybean oil need further research. With the improvements in analyses for metallic content, the time may be at hand for this study.

Metal inactivators effective in soybean oil (2,20,33,34) cover a wide range from sugar alcohols, such as sorbitol (5), to synthetic compounds having specific structures, such as carboxymethylmercaptosuccinic acid (23). The most universally added inactivator is citric acid. It can be used in the refining step (11) and in the initial and final stages of deodorization (13). When oil of good quality is processed in a plant where adequate control is maintained, addition of citric acid (0.005 to 0.01%) in the cooling stage of deodorization appears most desirable.

Figure 3 shows the substantial improvement in the product from laboratory deodorizations that can be achieved when citric acid is used properly. Coating the inside of a deodorizer (2) or using inert metal like polished stainless steel also helps keep metallic contamination low.

Some reports indicate that citric acid, phosphoric acid and certain other acidic materials are synergists for antioxidants in oil. Our studies suggest that with soybean oil citric acid acts exclusively as a metal inactivator (7). When soybean oil is exhaustively treated with sorbitol, a nonacidic metal inactivator, addition of citric acid gives no added improvement. This is true whether or not the metallic impurity or catalyst for oxidation was deliberately added or present originally. Figure 4 illustrates the effect of increasing amounts of sorbitol on 8-hr AOM peroxide values of soybean oil and of subsequent treatment with citric acid.

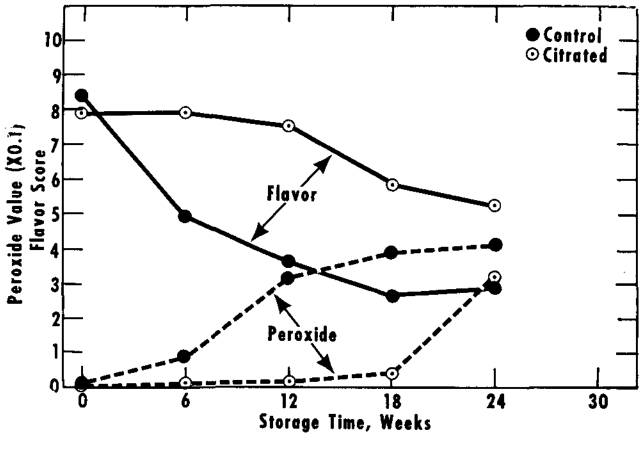

FIG. 3. Effect of citric acid on flavor and peroxide value of soybean oil during storage.

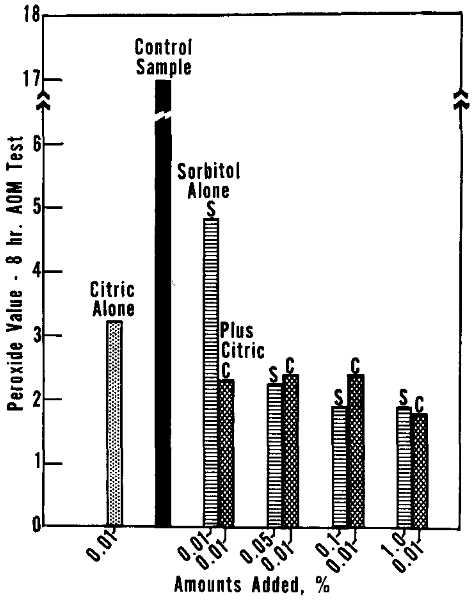

FIG. 4. Effect of added citric acid on the 8-hr AOM peroxide value of sorbitol-treated oil.

Another problem becomes important whenever soybean oils are mixed together or with other fats or oils. Often citric acid will not inactivate metallic impurities unless the oil is heated (3). Therefore, if a cool oil containing metallic impurities that were not properly treated with inactivator is mixed with soybean oil, the flavor and oxidative stability of the mixture can be substantially lower than that of either unmixed oil.

The effect of metallic impurities in untreated oil might not be particularly noticeable in use if the second oil contains no linolenate or other highly unsaturated ester and the amount of the metallic impurity is low. Some companies use citric acid or other metal inactivating agents when deodorizing any fat or oil.

Evaluating Oils

"The proof of the pudding is in the eating" and proof of the flavor is in the tasting. The best method available to measure quality of a salad oil is to taste it before and after controlled aging. Individuals can sometimes accurately judge flavor quality and stability of a number of samples at one time. If a group of people (a taste panel) is used, the subjective factor, i.e., individual bias and preference is reduced and more objective evaluations are obtained. A trained and thoroughly experienced observer in an industrial firm can be used for tasting or flavor evaluations but questions arise if there is a dispute between him and another expert. In contrast, when 8 to 12 people taste 2 or 3 samples, the taste panel operator can give a more nearly unbiased report on comparative quality and stability.

Taste panel members rate oils on an arithmetic scale and also describe flavor in specified terms. The numerical scores provide a flavor score; the descriptive words, a quality evaluation. Flavor scores are used to calculate standard deviations and significance of differences. Our taste panel members score an oil from 1 to 10 (highest quality) and describe the flavor that they taste. Tasters first arrange samples by odor alone so that the one with the best odor is first. They are asked to taste in this order. After the scores are averaged and compared, deviations and significances are determined (30,32).

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Key Factors in Flavor

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An important factor in testing stability is aging of the samples for tasting. Samples can be aged by a number of methods. We have selected exposure either in an oven heated at 60C for 3 or more days or to a fluorescent light for 1 to 6 hr. In oven aging, we use a bottle two-thirds full of oil, one-third full of air, and "closed" with a clean, loosely fitting, cellophane cover. In light aging, a similar half-filled bottle is placed in the center of a cylindrical chamber surrounded by six 15 in. 14-watt daylight fluorescent tubes and exposed for the necessary length of time (31). With soybean oil, this time is usually 1 to 2 hr. Heating to 150 or 200C and cooling is a more rapid aging test, and frequently some modification of this method is used in plant quality control.

The rapid oxidative test for stability that we use is a modification of the active oxygen method [Fat Stability, Active Oxygen Method—AOCS-Cd-12-57 (1)]. The sample is subjected to the conditions of the test (98C with wet air bubbling through the sample) for 8 hr. The peroxide value is determined and called the 8-hr AOM peroxide value. We also determine peroxide values on the samples that are aged for tasting by the panel: The higher the peroxide values, the less stable the oil; the lower these values, the more stable the oil.

Precursors of Flavor

Most research workers studying the flavor stability of soybean oil generally recognized that linolenate ester is the primary precursor of any unusual odors and flavors (i.e., odors and flavors that are different from those of cottonseed or other oils). One way to show that linolenate is a primary precursor is to react linolenate so that it becomes a part of glycerides of another bland oil and evaluate the odor and flavor of the product. In this fashion, cottonseed containing chemically introduced linolenate can be compared with soybean and cottonseed oil. When cottonseed oil has linolenate at the concentrations present in soybean oil, it is identified by the taste panel as soybean oil, not as cottonseed oil (10).

The combined effect of traces of copper or iron with oxygen appears to be associated with linolenate since the presence of metallic impurity and oxygen in the linolenate oil can accelerate the appearance of off-flavors. Other oils are affected by these trace metals and oxygen but linolenate and other oils containing more unsaturated esters are more sensitive. More will be said later about the effect of air. The particular part of the linolenate molecule that is so important to flavor is believed to be $CH_3CH_2CH{=}CHCH_2$- or its isomeric products (9,15).

Studies show that, with exception of the nonpolar fraction of the unsaponifiables of soybean oil, other minor components are not precursors. Squalene appears to be the nonpolar component most likely to cause some flavor problems, but its effect compared to metals, air and linolenate is believed to be small (17,25). Addition of 0.1 to 0.2% of squalene to soybean oil lowers the flavor score of the aged oil as compared with flavor scores of samples without added squalene.

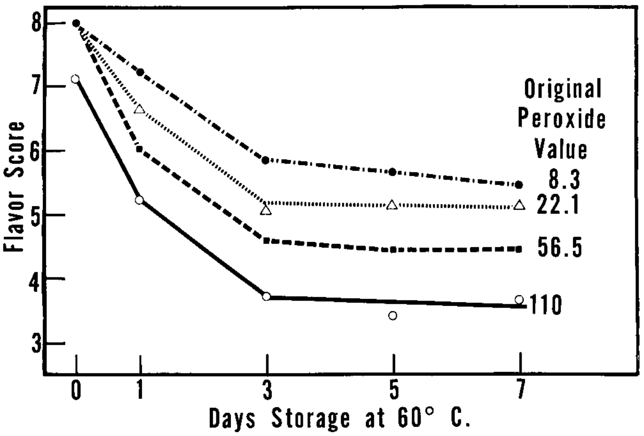

FIG. 5. Effect of autoxidation before deodorization on the flavor and oxidative stability of soybean oil.

TABLE I

Effect of Dissolved Air and Light on the Flavor Stability of Soybean Oil [a]

Condition	Initial flavor score	Aged flavor score	Increase in peroxide value	Sig.
Clear glass bottle—aged at 60C in the dark for 4 days				
Open to air	8.1	3.7	+27.4	**
Nitrogen packed	8.1	7.4	− 0.3	*
Two-thirds full (sealed with air)	8.1	4.8	+ 5.5	**
Full (sealed with air)	8.1	6.4	+ 1.2	*
Brown glass bottle—exposed to fluorescent light for 2 hr				
One-half full	8.1	5.5	+ 1.8	**
Full	8.1	6.7	+ 0.4	**
Nitrogen packed	8.1	7.6	+ 0.3	*

[a] Key: *, odds are 20 to 1 or better that the difference is real; **, odds are 100 to 1 or better that the difference is real.

Recent Developments

Three fairly current developments show that substantial improvements in soybean oil can still be achieved in research and commercial operations. The first of these developments represents an engineering and processing achievement that is based on a number of "bits" of information that we have published. The product might best be called "protected" oil and the process, an integrated one. This development has resulted from engineering application of research information on the effect of metals, citric acid, bleaching, air, nitrogen protection and the lowering of tocopherols, and how large-scale treatment or refining, bleaching and deodorization should be carried out. A brief review of the published research information may be helpful.

Enough air can dissolve in an oil and react to give a peroxide value of about 18. Of course, some peroxides may

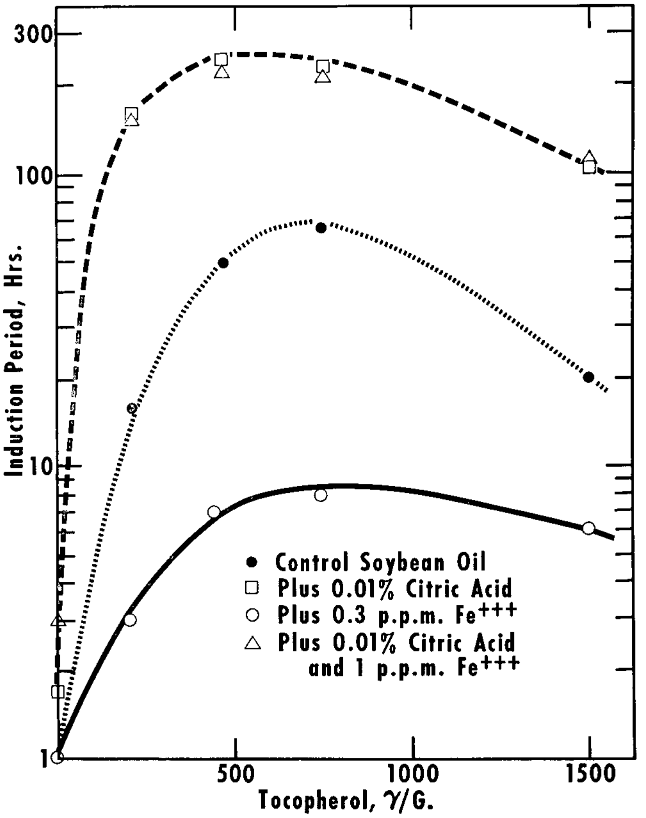

FIG. 6. Effect of citric acid and iron at different tocopherol levels on oxidative stability.

TABLE II

Comparison of Bleached and Unbleached Protected Soybean Oil

Condition	Flavor score (peroxide value)	
	Bleached	Unbleached
Initial flavor	7.9(0.8)	6.7(0.5)
Aged flavor	6.4(1.8)	5.2(4.0)
8-hr AOM peroxide value	2.2	20.0

decompose before all of the oxygen reacts. Light and heat can accelerate this reaction with oxygen. Table I contains data on the effects of dissolved air, of light and of protecting an oil with nitrogen. Note that protection of oils either with nitrogen or by storage in brown bottles was excellent but a combination of both protective methods gave best flavor scores.

Exposure of the oil to air and metals after alkali refining and before deodorization also adversely affect stability. This debilitation can be expected on the basis that oxidation of an oil before deodorization clearly affects stability (21). Figure 5 shows the effect of initial autoxidation to peroxide values of 8, 22, 56, before deodorization on the change in flavor scores and peroxide development when the deodorized samples are aged in an oven at 60C: The higher the peroxide values before deodorization the more rapid the oxidations and the loss in flavor scores on aging. When oxidative polymers are isolated from the oxidized oil and when these are added to samples of high-quality soybean oil, flavor stability is significantly lowered (21). This result is further proof that oxidation before deodorization is an important factor in stability. Protection of the oil from the alkali-refining step through packing and use is desirable. This need for protection is characteristic of all oils but is more readily discerned with oils or fats containing polyunsaturates, such as linolenate.

When the metals are properly inactivated with citric acid or other metal inactivator, soybean oil can contain too much natural tocopherols. Figure 6 shows that a decrease in tocopherol content from 1500 to about 600 μg/g increases the induction period for the control soybean oil even when iron alone is added to the sample (24). Apparently the higher amounts of tocopherol serve as prooxidants.

Although bleaching earth can add undesired metallic impurities, bleaching still increases the stability if the metallic traces are inactivated and if the oil is protected from air. Vacuum bleaching and possibly nitrogen blanketing appear desirable in this operation. Table II compares the flavor quality and stability of two soybean oils prepared in the same commercial plant. The bleaching step was included for one sample but omitted for the other (6).

Some engineering concerns and oil refiners have put into operation the necessary engineering and quality control procedures that give improved salad and cooking oils without hydrogenation. Indeed, we have found that some of these oils are equal, or superior, to hydrogenated-winterized soybean oil (HWSB) in most flavor stability tests except those based on frying potato chips and on heating. Data that permit direct comparison of a protected oil and HWSB oil are given in Table III. Recent samples of protected oils did have better 8-hr AOM peroxide values with some samples as low as 1.5. When protected oil was used to fry potato chips, initial quality of the chips was good. Aged chips fried in HWSB oil, however, were superior to aged chips fried in protected oil (6), as shown in Figure 7. A later test shows that addition of an antifoaming agent to the oil improves the quality of the aged potato chips that were fried in the "protected" oil. Further pilot tests are in progress.

TABLE III

Comparative Values for Hydrogenated-Winterized Sybean Oil and Protected Oil

Condition of aging	HWSB Oil	Protected oil
None		
Flavor score	8.3	8.1
Peroxide value	0.3	0.4
Aged 60C for 4 days		
Flavor score	7.1	6.5
Peroxide value	1.4	1.6
Light exposure, 2 hr		
Flavor score	4.7	5.8
Peroxide value	2.2	3.7
Heating to 300F		
Flavor score	5.3	4.8
Peroxide value	0.6	1.3
8-hr AOM		
Peroxide value	2.6	3.3

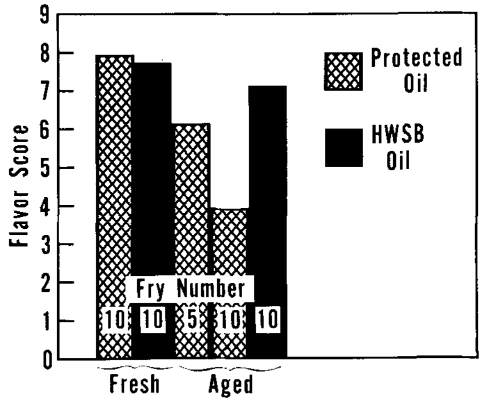

FIG. 7. Flavor scores of fresh and aged potato chips fried in HWSB oil and in "protected" soybean oil. Both oils contained butylated hydroxyanisole (BHA) and butylated hydroxytoluene (BHT) and citric acid. The "protected" oil did not contain an antifoaming agent.

Applying some of the information about protected oil coupled with that on hydrogenation-winterization has led to the second recent development. HWSB oil is easy to describe in terms of the operational procedures used to make it (18,29): hydrogenation, winterization, filtration to remove solids and any catalyst residues, deodorization and addition of inactivators, antioxidants and antifoam agents. Practically, it is necessary to exercise proper quality control over these operations to maintain quality of the final product. Laboratory studies have shown that undue exposure to air and metals or some unknown factors lower flavor stability of HWSB oil as compared with the control. Citric acid treatment of the oil shows that metallic contamination is one factor involved because the citric acid does improve laboratory HWSB oil more than laboratory control oil (4). This contamination can occur with hydrogenated oils too, but hydrogenation-winterization involves more handling and possible exposure both in laboratory and plant. Thus, oxygen and metallic impurities may have greater opportunity to exert their adverse effect before deodorization in the case of HWSB oil than of an unhydrogenated or hydrogenated oil.

Some qualitative evaluations of an HWSB oil are compared with those of another commercial soybean oil made in 1962 (Table IV). The most significant improvement found for HWSB oil is in the quality of food fried in it as compared with food fried in unhydrogenated soybean oil of good quality (commercial 1962). Figure 8 shows that flavor scores of potato chips from the first fries were acceptable, but as the potato chips came from the fifth or later fries and were aged, flavor scores of potato chips from HWSB oils were significantly higher (5).

The third development is still in laboratory and pilot stages. Catalysts for hydrogenating soybean oil have been found that have high selectivities for reducing linolenate compared with linoleate. The nickel catalysts now used have rather low selectivities for linolenate over linoleate. Se-

TABLE IV

Comparisons of Hydrogenated-Winterized Soybean Oil and Soybean Oil (Commercial Oil)

Condition	Peroxide value for		Remarks
	HWSB oil	Soybean oil	
Initial flavor	8.3(0.3)	7.6(0.9)	Stored at OC
Aged flavor	7.1(1.4)	3.8(2.4)	60C for 4 days
Heating to 300F	6.3(1.1)	5.0(3.0)	Rapid heating
Light exposure, 2 hr	4.7(2.2)	4.6(2.8)	Special apparatus
8-hr AOM	2.6	23	98C–air

TABLE VI

Effect of Hydrogenating with Copper Chromium Oxide Catalyst on the Flavor Stability of Soybean Oil

	Hydrogenated with Cu-Ba-Cr catalyst		
	Treated with citric acid	No citric acid	Sig.[a]
Initial flavor score	7.7	6.7	*
Aged flavor score[b]	6.8	4.8	**
Aged peroxide value	0.7	6.2	
8-hr AOM peroxide value	4.5	95	

[a] See Table II.
[b] Aged 4 days at 60C.

lectivity is defined here as the ratio of the rate for reducing linolenate divided by the rate for reducing linoleate ($S = K_{Le}/K_{Lo}$). The most active of the catalysts are prepared by reducing copper and copper-chromium salts to their metallic oxides with such agents as sodium borohydride (27,28). When hydrogenations are carried out with 1 cc of oil and if linolenate and linoleate are determined by gas-liquid chromatography (GLC), the selectivity for linolenate over linoleate is 6.0 to 7.3. Selectivity for the same oil with nickel and nickel-modified catalysts ranges from 1.7 to 2.2. When linolenate is measured by alkali isomerization, selectivities for linolenate over linoleate in runs with 300 g of oil vary from 9.5 to 13 (28). Selectivities calculated from alkali-isomerization data are usually higher than these based on GLC since the hydrogenation produces a small amount (about 1.0%) of conjugated diene, which elutes with linolenate in GLC analysis and gives high values for linolenate. Less than 1% linolenate is left in the oil. The selectivities for linoleic over oleic approach infinity since no readily measurable increase in saturates occurs. Table V gives the conditions used and the selectivities and compositions achieved with different catalysts (27,28).

Again, the proof of the pudding is, of course, in the eating; the problem is not whether the linolenate is selectively hydrogenated. Although preliminary results show some effects that most observers would expect from exposure to copper, our evaluations are nevertheless encouraging. Metallic impurities, probably copper, left in the oil are powerful prooxidants; however, a significant favorable change does occur. Flavor results after storage at 60C for 4 days gave rancid responses predominantly. The flavor responses at low levels of autoxidation in the presence of metallic impurities were not characteristic of soybean oil. Also, citric acid treatment did substantially improve both flavor and oxidative stability as shown by 8 hr-AOM tests (peroxide values) and flavor scores after aging at 60C for 4 days in the presence of air (16). Our results are summarized in Table VI and they should encourage anyone familiar with the problem of stabilizing soybean oil to do more work. Cold tests on some of the soybean oils reduced with copper-chromium oxide catalysts gave no precipitate or clouding. Further studies are needed to achieve the objective of commercial production of an improved soybean oil by selective reduction and elimination of linolenate. Catalytic hydrogenation without loss of linoleate and without formation of *trans* isomers is still an unsolved objective of our research.

Summary

Our work shows that linolenate, metallic impurities, metal inactivating agents, air and light are important factors in the flavor stability of soybean oil. Processing techniques, such as hydrogenation-winterization, deodorization at high temperatures and low pressures, protection with nitrogen, and the use of copper-chromium-oxide or newer copper oxide catalysts, have led or may lead to better oils for consumers. Although more modern equipment, such as stainless steel deodorizers and improved devices for use of nitrogen and steam, is highly desirable, older plants that have large-batch steel deodorizers still can make a good quality oil. Quality control is necessary with both modern stainless or older steel equipment to make soybean oil of good flavor and stability.

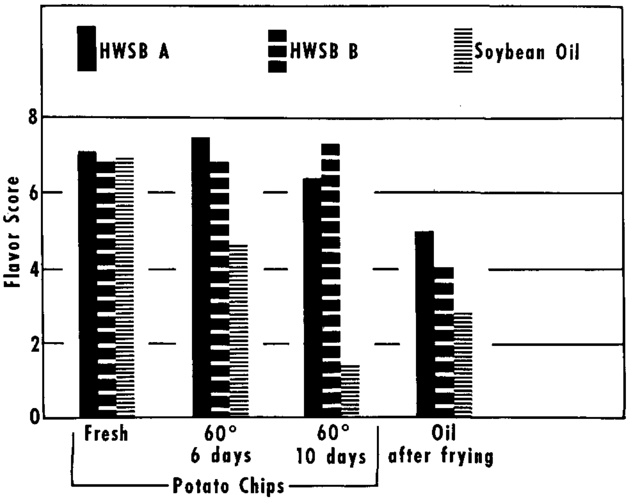

FIG. 8. Flavor score of potato chips fried in HWSB oil and unhydrogenated soybean oil. Chips fresh and aged.

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TABLE V

Comparison of Catalysts for Selective Hydrogenation of Linolenate—$K_{Le/Lo}$

Catalyst	Hydrogenation			Linolenate % (triene)[a]	Linoleate % (diene)[a]	*trans*[b] %	Selectivity $K_{Le/Lo}$
	Pressure	Temperature	Time, min				
Original oil				7	47	0	
CuCr ($NaBH_4$)	30	170C	63	0.4	37	9	12
CuCr ($NaBH_4$)	30	200	17	0.7	42	8	10
CuCr (Commercial)	30	170	65	0.3	40	9	12
Copper ($NaBH_4$)	30	200	18	0.5	41	12	10
Nickel	8	140	17	3.3	30	12	1.9

[a] Analysis by alkali isomerization.
[b] Infrared analysis.

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• *Local Section News*

Northern California Section

C. W. Hoerr Addresses Spring Meeting

On Friday, April 29th, the Section held its Spring Meeting at Louie's Restaurant of Grant Avenue. Following the social hour and an excellent Cantonese dinner, C. W. Hoerr, Administrative Assistant, R & D, Durkee Famous Foods, Chicago, spoke on the subject "Applications of Fat Crystallography in the Food Field." As newly elected President of the AOCS, Mr. Hoerr also talked about the program of the National Society. The question and answer session indicated a keen interest in fat crystallography. The Section was pleased to also have as guests, Mr. Hoerr's wife, Verna and their son Bill and daughter Leslie. The meeting attendance was 67, which represents a good percentage of the membership, and one of the highest attended.

Fall meeting in Fresno, California.

Plans are being made for a two-day meeting in Fresno, California, at the Hacienda Motel, on October 21–22. The program will include speakers and plant tours to Ranchers Cotton Oil Co., and Producers Cotton Oil Co. Details will be announced later.

• *Industry Item*

Photovolt Corporation of New York City, supplier of scientific laboratory instruments, has recently been acquired by Bio-Science Laboratories of Van Nuys, Calif. Development activities of Photovolt are expected to be broadened with the help of the extensive scientific staff and the research programs of Bio-Science. There will be no management or policy changes at Photovolt Corporation.