• *Bailey Award Address*

Twenty Years of Research in Oils at Northern Regional Research Laboratory[1]

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Research conducted at the Northern Laboratory on vegetable oils during the past two decades includes polymerization, isomerization, flavor stability, and structure. Some early accomplishments—dimeric fatty acids and their derivatives, conjugated oils and their aftertack as well as the use of citric acid to inactivate iron in soybean oil—and more recent accomplishments—aldehyde oils, cyclic fatty acids, and tests for hidden oxidation and hydrazine reduction—are among the subjects reviewed.

Research in fats and oils in the U.S. Department of Agriculture began shortly after the Bureau of Chemistry was formed in 1906. Jamieson, author of a book revised in 1954 by E.W. Eckey, was a leader in this early research.

The U.S. Regional Soybean Laboratory was established in 1936 under the Bankhead-Jones Act. Milner, Wheeler, Hopper, and Markley, names well known to you, were leaders at the Urbana Laboratory, and the first two of them continued their careers for a period at Peoria. Congress created the Regional Research Laboratories in 1938, and the executive branch of the government located the Northern Laboratory at Peoria, Ill.

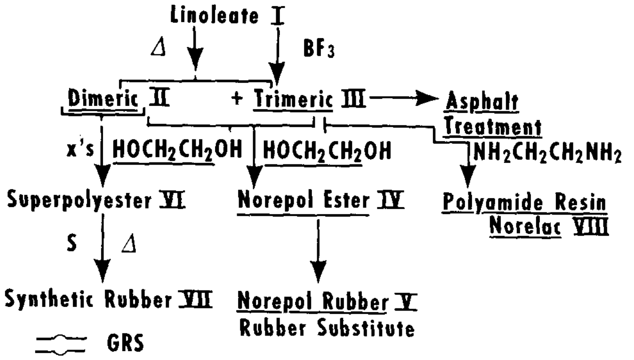

FIG. 1. Preparation of dimeric and trimeric acids and their polymeric derivatives (underlined products have been or are commercially available).

Research on fats and oils at Peoria was initiated in the summer of 1940. I fully appreciate that my selection for the Bailey Award encompasses the varied aspects explored by oil chemists on our staff and the many contributions made by them. More than 350 papers and patents have resulted from our studies on fats and oils. Reading the names of the authors and the titles of these publications would consume more space than we have. After careful consideration I have chosen to write primarily on four areas of our work: polymerization, isomerization, flavor stability, and structure.

Polymerization

Prior to 1940 industry practiced the art of bodying oils to make varnishes and enamels. Some knowledge of how oil molecules reacted with one another was available. The composition and structure of the products were unknown although some predictions on their structure had been made. In 1941 Bradley reported the separation of dimeric and trimeric fatty acids (1).

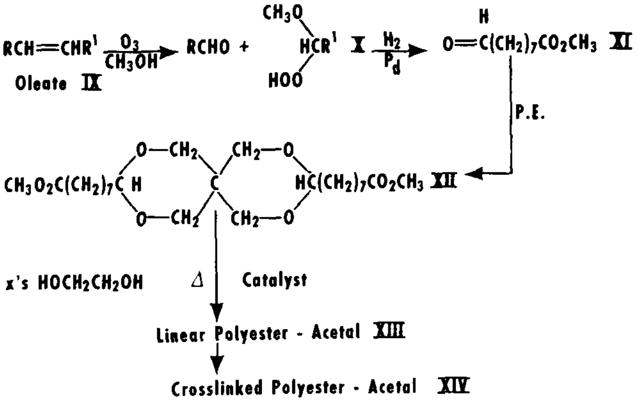

FIG. 2. Preparation of azela-aldehydate and crosslinkable linear saturated polyester.

Dimer Acid and Its Derivatives. Today dimer acid can be purchased as a commercial chemical, and we believe that our efforts helped to effect its commercialization. In Figure 1 linoleate I (also linolenate and other unsaturated esters) is shown to give on heating, with or without catalysts, dimeric II and trimeric products III. We found that mixtures of these esters could be reacted with ethylene glycol to give a sticky polyester IV that resembled natural rubber in some properties (2). By reacting this polyester with sulfur or sulfur compounds or by further polyesterification under reduced pressure, a millable product was formed. Some of you will remember the trivial name we gave it, Norepol, after Northern Regional polymer. Much simpler, you will agree, to say than the vulcanized polyester from polymeric fatty acids and ethylene glycol. It was a rubber substitute, not a replacement, and a recap tire made of Norepol rubber V ran 1,500 miles before it failed. Purification of dimeric ester and proper reaction with ethylene or other glycol gave a superpolyester VI (3). Although simply said, it was not simply done. To make the polyester with ethylene glycol, it was necessary for us to discover the same techniques of excess glycol and the glycolysis reaction of polyester formation used for making Dacron without any knowledge of how Dacron was made. On vulcanization of the superpolyester, the product, a synthetic rubber VII, had properties similar to the then available vulcanizate GR-S, *i.e.,* butadiene styrene copolymer (4). Thus we were able to convert soybean oil to a rubber substitute and to a synthetic rubber. Indeed some 2 million pounds of Norepol products were manufactured and used during the latter part of 1942 and early 1943. Our publications were considerably delayed because of government secrecy orders.

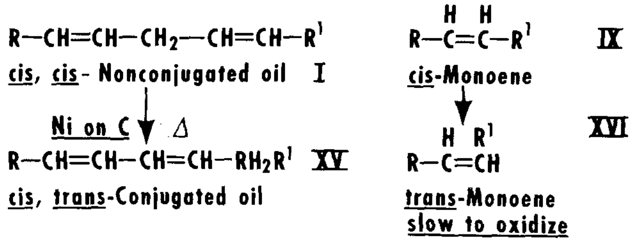

FIG. 3. Isomerization of linoleate and oleate.

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[1] A.E. Bailey Award Address before meeting of North Central Section of the American Oil Chemists' Society, Chicago, Ill., March 29, 1961.

[2] This is a laboratory of the Northern Utilization Research and Development Division, Agricultural Research Service, U.S. Department of Agriculture.

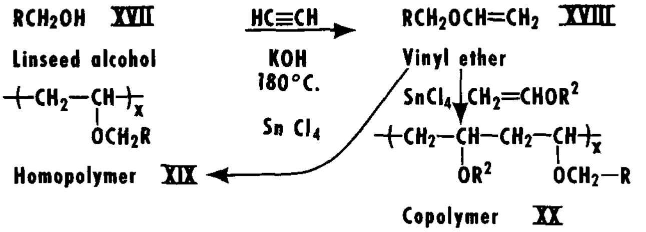


FIG. 4. Preparation of vinyl ether polymers from linseed fatty alcohols.

One feature of our rubber work was the use of di-isocyanates to vulcanize and crosslink the polyesters to give another route to rubberlike products (5). In closely related work the dimeric fat acids were reacted with diamines and amino alcohols to give polyamides and polyesteramides. Probably the most interesting of these polymers was the ethylenediamine polyamide, then called Norelac VIII (6), now sold commercially by General Mills and other companies. These polyamides are useful for many products, but their first commercial use was in an unexpected place, unexpected to us. Melting at 116°C., flexible, and adherent to most surfaces, these resins found their first sustained commercial use as a heat-sealing resin for glassine paper in wrappings for chocolate candy bars. As many of you know, other market items have been found for these resins, such as gel paints (7) and as adherent, flexible coatings for concrete (8). Initial experiments were made on these polyamides to find a polymeric product that could serve as a war-time protective coating in place of restricted tung oil. I suppose this result proves that you must follow where your research leads you. To fail to do so means you may miss important discoveries that you might otherwise find. Good, intelligent research, either basic or applied, does not always follow a predetermined course. Often when you try to put research in a cubby-hole, it jumps out some place else.

Trimeric Fatty Acids. As a basic ingredient for linear polymers, trimeric fatty acids are not desirable, but as crosslinking agents in condensation polymers they are unique. They have good low-temperature properties, including flexibility and water repellency. An easy way to make them is to use HF and BF_3 catalysts (9). It is our understanding that large amounts of these trimers may soon go into the construction of bridge coverings and airport runways.

One measure of success in research on a given subject can be gauged in part by the number of other people that use your publications as stepping stones, *i.e.,* base new work on yours. Usually you do not have any easy way of counting all of the publications that follow later. We were fortunate because a commercial concern counted them for us. Since our early publications on uses for dimer acid more than 150 publications and patents have been published, a ten-fold increase.

Aldehyde Oils. In the past few years we have undertaken a comparatively new line of basic research on aldehyde oils. Only a few of our publications have been issued, but several will appear soon. However our exploratory studies conducted with methyl oleate will show some chemical changes that can be effected (10). Methyl oleate, IX in Figure 2, is ozonized in the presence of a reactive solvent, such as methanol, to give aldehydes and methoxy hydroperoxides X. By use of the proper catalyst and hydrogenation conditions, the pelargonic aldehyde and azela-aldehydate XI are obtained. Now these two products are both active, and any good chemist can write many reactions that he might carry out.

TABLE I

Vinyl Ether Film Properties

Baked 450°C. for 15 min. Drier 0.01% Co–0.5% Pb

Polymer film	% Styrene	Hardness (Sward)	Caustic resistance (hrs.)	Xylene resistance (hrs.)
IB (3) CS (1)	....	11	<3	<3
CP (1.5) CL (1)	....	50	>216	>144
CP (1.5) CL (1)	15	....	>888	Poor
DCP (3) CL (1)	40	....	>888	Poor

IB, isobutyl; CP, cyclopentadiene; DCP, dicyclopentadienyl; CS, conjugated soybean; Cl, conjugated linseed

One reaction we have studied is with the polyol, pentaerythritol. A new dibasic acid is formed. Its name by Geneva rules is a real hazard in any talk, so we chose to call it the pentaerythritol acetal of azela-aldehydate XII. Here our experience with ethylene glycol-dimer acid polyesters was valuable. When the free acid of XII reacts with ethylene glycol to give a polyester, the product is insoluble and infusible, *i.e.,* a crosslinked polyester. If the polyester is formed by a glycolysis reaction, a linear superpolyester-acetal XIII can be obtained. Add to XIII a trace of acid at an elevated temperature and a crosslinked polyester XIV results that is transparent, tough, and strongly adherent to glass surfaces. This type of linear polyester and the method of crosslinking the polyester are new advances in the basic chemistry of vegetable oils and polymeric products (11).

Isomerization

One objective of oil chemists in the 1940's was to convert a nonconjugated drying or semidrying oil, such as soybean or linseed oil, to a conjugated oil, such as tung or oiticica oil. We were among those who spent some time on this problem. We were fortunate to be associated with Peter Kass, who initiated studies in this country on the alkali isomerization of linoleic and linolenic acid (12). These and other studies led to better methods of analyzing for fats and to a search for other methods of isomerizing soybean, linseed, and other oils to give conjugated oils. Some of our work on conjugated oils and aftertack is summarized in Figure 3. Our studies led to a nickel-on-carbon catalyst that was very effective in giving comparatively large amounts of conjugated fatty products (13). Thus I could be readily converted in 50–70% yields of conjugation, based on starting diene. Catalysts similar to it have been used effectively in commercial operations in Europe to give conjugated linseed oil (14). However, when the conjugated oil is processed into varnishes and enamels, most, but not all, of the advantages of the introduced conjugation are lost (15).

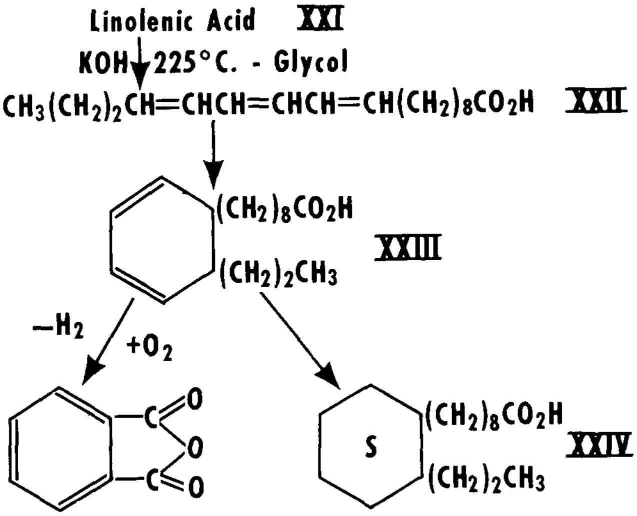


FIG. 5. Preparation of cyclic fatty acids.

You will remember that these oils had another disadvantage. Some of them developed "aftertack," *i.e.,* some of the films of their products dried rapidly to tack-free products but one to two weeks later became sticky and tacky, *i.e.,* aftertack. Our studies on the chemistry of isomerization gave us a logical explanation, a hypothesis, for this phenomenon. Conjugating agents move one double bond to form *trans* bonds in a new *cis-trans* conjugated diene. Inevitably some isolated *trans* bonds are formed. *Cis*-unsaturated fatty acids, such as oleic acid IX, react fairly rapidly with oxygen in the presence of a diene and drier to give polymers. *Trans*-unsaturated fatty acids, such as elaidic acid XVI, do not. Thus, in a film containing drier catalysts, a *cis*-monounsaturated fatty acid participates in initial film formation, but a *trans* acid does not. However, after the film has formed, the *trans* acid can still oxidize but much more slowly, and the "volatile" products of its oxidation cannot escape so readily. These volatile products remain to plasticize the film and give aftertack (16). Calcium oxide in the film reduces this aftertack (17).

Vinyl Ethers. More recently we have engaged in studies involving rearranged fatty products that are derived from alkali-isomerization. In Figure 4 are shown the reactions that convert a fatty alcohol, such as linseed alcohol XVII, to the corresponding fatty vinyl ether XVIII. By treatment with $SnCl_4$, the vinyl ether reacts to give a polymer (18). The homopolymers XIX fail to air-dry and convert to a useful film as one might expect from their functionality, *i.e.,* molecular weights of 5 to 10 times that of linseed oil, with only "slightly" modified functionality in the fatty chain. When copolymerized with isobutyl vinyl ether to give a copolymer XX (where R^2 is isobutyl), some improvement in film is achieved. Teeter and others have found that allyl vinyl ether (19), dicyclopentadienyl vinyl ether, and other monomers (20) with bulky groups give copolymers with improved film properties. Table I shows the extent of this improvement in air-drying, hardness, and alkali resistance of the baked films that is achieved by "cyclic" monomers copolymerized with linseed vinyl ethers (21). Styrenation improves the properties of these polymers by improving air-drying, baking, and other film characteristics, such as hardness and alkali resistance (22).

The fatty alcohol appears to have its structure more than slightly changed when linseed fatty alcohol is used. This work leads directly to the next study.

Cyclic Fatty Acids. A few years ago we investigated dibasic acids that could be obtained by alkali-isomerization, hydrogenation, and oxidative cleavage (23). This work with the dibasic acids and the vinyl fatty ethers reminded us of unpublished work at our laboratory by Skell, Kass, and Radlove (24). Accordingly we investigated and found that alkali-isomerization of linolenic acid gave high yields of a cyclic fatty acid (25). Eleostearic and other conjugated trienoic acids, or acids that readily form conjugated trienoic, will do likewise in the presence of alkali and of alcoholic or glycolic groups at elevated temperatures. Apparently (Figure 5) the alkali at elevated temperatures converts linolenic acid XXI to a *trans,cis,trans*-conjugated triene XXII that cyclizes to a cyclohexadiene XXIII. Presence of a six-membered ring was confirmed by conversion to phthalic anhydride. Of considerable interest is the noncrystalline hydrocyclic acid XXIV that has a very low pour-point (−40°C. or lower) (26).

Flavor Stability of Soybean Oil

When W.H. Goss of our staff went to Germany in 1945 for the U.S. Technical Industrial Intelligence Committee, his report (27) indicated that a variety of procedures improved the flavor stability of soybean oil. One of these procedures was the addition of 0.01% citric acid to the oil. Although citric acid was accepted as an "acid synergist" for antioxidants at that time, its mode of action as a metal-inactivating agent was obscure.

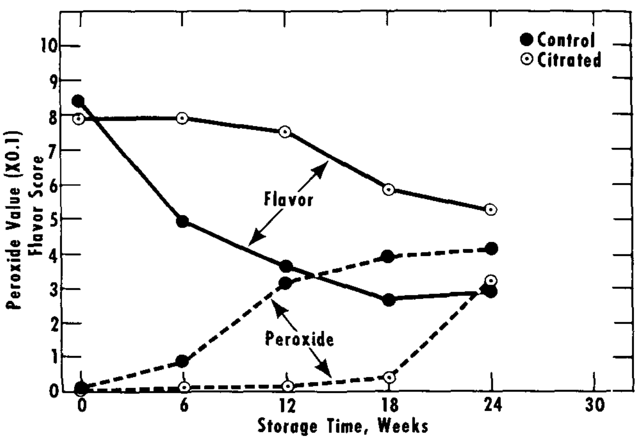


FIG. 6. Changes in flavor score and peroxide value of "citrated" and "uncitrated" samples on storage at room temperature.

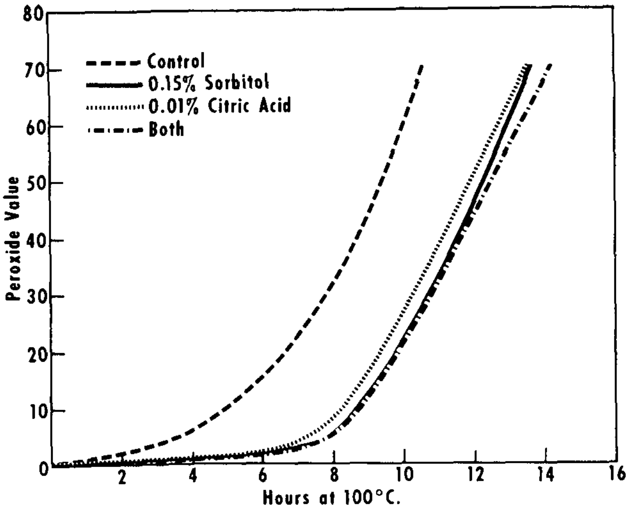


FIG. 7. Stabilizing effect of either 0.15% sorbitol or 0.10% citric acid and the combined effect of 0.15% sorbitol and 0.005% citric acid in soybean oil oxidized at 100°C. under Active Oxygen Method conditions.

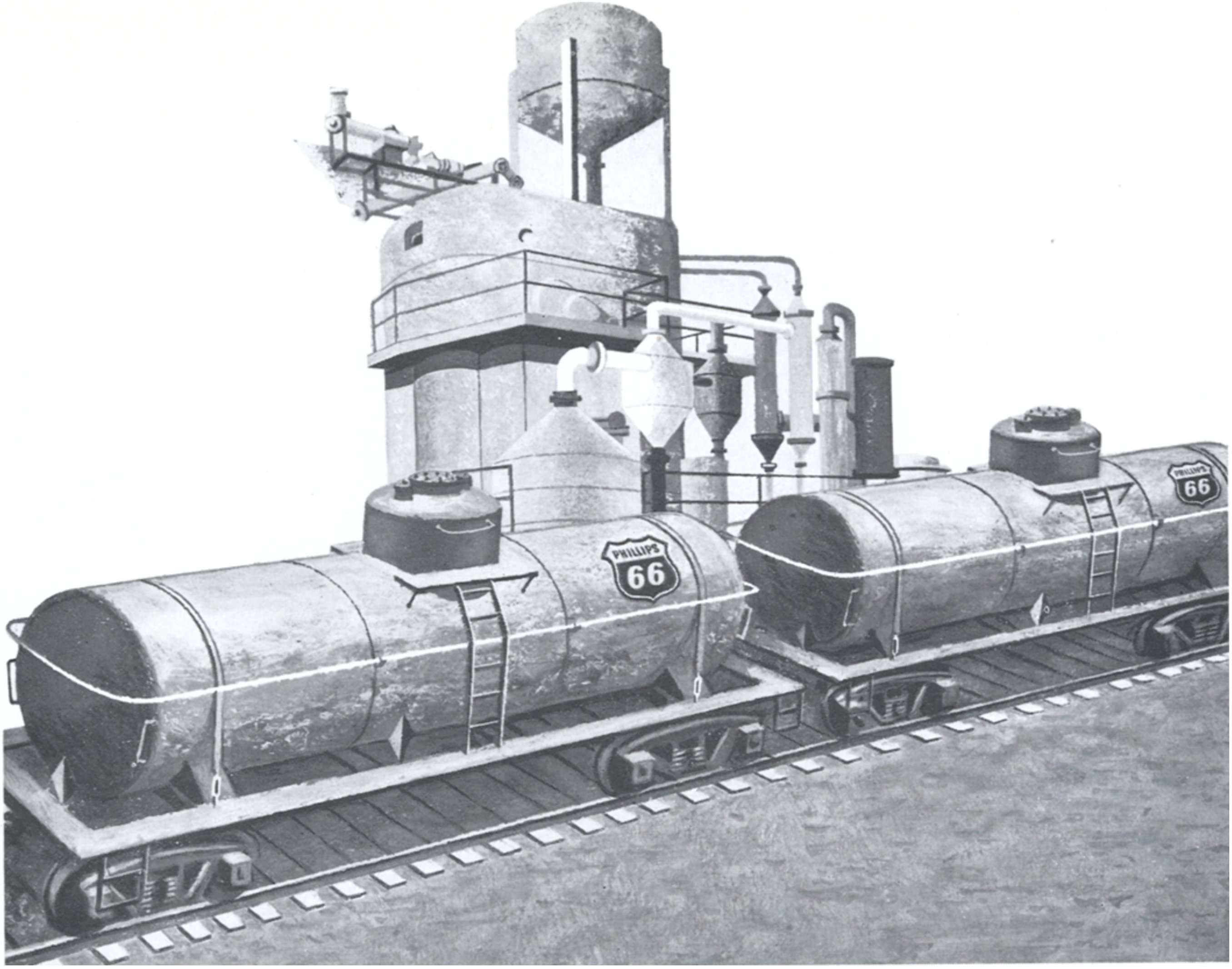
Citric Acid Improves Soybean Oil. In Figure 6 is shown the dramatic improvement that proper use of citric acid can bestow on soybean oil (28). After repeated laboratory trials and investigation of other reported procedures for improving soybean oil, it was clear to us that proper use of citric acid or related products was the most important of the many German procedures that merited immediate commercial trial. Through the cooperation of Armour and Company and others we were able to make several large-scale trials.

Alas, they were initially a failure insofar as obtaining an improved soybean oil was concerned. These failures caused some scratching of heads and wonderment. Careful consideration of these pilot-plant trials led to still further work, which showed conclusively that, in commercial equipment, citric acid did improve the flavor stability of soybean oil (29). Citric acid inactivated trace metals present in soybean oil (30). Unbelievably small amounts of metal lowered the stability of soybean oil. Our research showed that 0.1 p.p.m. of Fe and 0.01 p.p.m. of Cu were sufficient to lower the stability of soybean oil (31). In the commercial and pilot-plant trials that failed, the amounts of iron or copper present or introduced into the oil were too great for the amount of citric acid added to inactivate the metals.

For some time the acid synergist nature of citric acid has been in question. The improvement in soybean oil and lard effected by sorbitol, a nonacidic metal-inactivating agent under conditions of maximum efficiency, is great enough to render the addition of citric acid ineffective (Figure 7). Thus the action of citric acid in soybean oil is established primarily as a metal-inactivating agent not as an acid synergist (32).

Following these discoveries, numerous metal-inactivating agents were tried and found effective (33). However an accepted fat-soluble, nontoxic, heat-stable, effective metal-inactivating agent is still not available. Here is a research problem worthy of consideration by any of you.

Concurrently with, and after, our elucidation of the action of citric acid and metals in the flavor stability of soybean oil, the use of soybean oil increased from a figure of 1 billion pounds to more than 3.1 billion pounds today. Cold rolled steel deodorizers were in vogue in 1946–48; today stainless trays for deodorizing the soybean oil are accepted equipment in the construction of deodorizers. Stainless steel adds little or no additional metal to oil. However the oil contains sufficient trace metal, when processed through a modern plant even equipped with a stain-



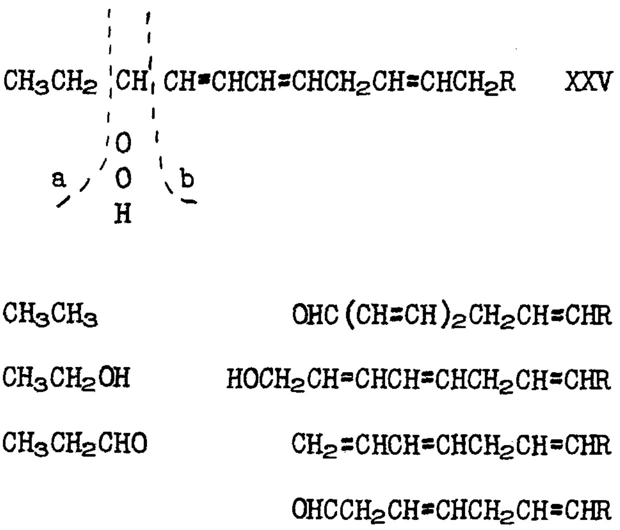


FIG. 8. Products from primary decomposition of methyl linolenate hydroperoxide.

less deodorizer, to warrant treatment with citric acid or other inactivating agent. Now we do not believe that we were solely responsible for all these commercial changes, but we do believe our work brought these changes much sooner and more quickly than might otherwise have occurred.

Linolenic Acid a Precursor of Flavors. The cause for flavors in soybean oil has been blamed on a variety of components, such as linolenic acid, phosphatides, and unsaponifiables. Some years ago we conducted experiments that confirmed linolenic acid as a primary precursor of flavors (34). Its removal in soybean oil by hydrogenation imparts high stability, as all of you know from the use of margarine or shortening now made largely from soybean oil. To obtain the special oils mentioned in Table II, linolenic acid (Le) and linoleic (Lo) acid were tranesterified into cottonseed oil to give two different oils. Our taste panelists were unable to distinguish the linoleic acid-cottonseed sample from cottonseed oil but identified the linolenic acid-cottonseed oil as soybean oil.

TABLE II

Number of Identifications on Stored Samples as Cottonseed Oil (CSO) or Soybean Oil (SBO) of Cottonseed Interesterified with 7.5% Linoleic (CSO-I Lo) or 7.5% Linolenic (CSO-I Le)

Sample	CSO	SBO	Neither	Sig.[a]
SBO	0	6	0	
CSO	5	1	0	‡
CSO-I Le	1	5	0	
SBO	1	7	0	
CSO	7	1	0	‡
SBO	2	6	0	
SBO	0	7	0	
CSO	7	0	0	‡
CSO	7	0	0	
SBO	0	7	0	
CSO	6	1	0	‡
CSO-I Lo	6	1	0	

[a] ‡ Highly significant difference; odds are greater than 100–1 that the identifications are correct.

The possibility that another factor is involved is clearly shown in the work on unsaponifiables (35). Under a fellowship grant from the Soybean Council of America Inc. we have recently undertaken work on the "other factor." We have confirmed the earlier work, but the identity of this factor remains uncertain (36). Our conjectures about this unsaponifiable factor might surprise you, but we must wait until we have confirmed or disproved them before disclosing our present findings on the unsaponifiable fraction.

Autoxidation and Flavor Quality. Autoxidation is, in our opinion, the chemical process that produces flavors in soybean oil and its products. With soybean oil samples of known history and treatment, the quality of the oil can be predicted by its peroxide value. A correlation coefficient of −0.87 was found between the logarithm of the peroxide value and the flavor score. Let me emphasize that this correlation is highest with oils of known history and oxidized under controlled conditions (30). Studies of ours and of others show that a host of carbonyl compounds do result from the autoxidation of soybean oil and linolenic ester. Table III lists sme of these compounds that come from both linolenic acid (37) and soybean oil (38). More recently, using the latest techniques of gas chromatography and mass spectrography, we have confirmed the presence of some of these products and have found a host of new ones. If you wish an exercise in the chemistry of fatty acids, list the predictable compounds that you can expect by the cleavage of hydroperoxides from linolenate esters as shown in Figure 8.

TABLE III

Products from Soybean Oil (S) (38) and Linolenic Acid (Le) (37)
Volatile cleavage products

CH_3CHO	S + Le
CH_3CH_2CHO	S + Le
$CH_3CH = CH\ CHO$	S
$CH_3CH_2CH = CH\ CHO$	S + Le
Hexen-dial	Le
Hexanol	S
$CH_3(CH_2)_3CH = CH\ CHO$	S

Linolenate hydroperoxide XXV cleaves initially in a variety of ways so that at least seven different compounds are formed, depending on whether or not the molecule breaks at a or b. Only one hydroperoxide and none of the secondary products derived from the cleavage products are shown here. In the March issue of the Journal of the American Oil Chemists' Society we reported on the isolation of aldehydo esters as well as alcohols and hydrocarbons (39). Thus cleavage does occur on either side of the hydroperoxide in a variety of ways. If you undertake the exercise on the cleavage of hydroperoxides, please remember that there are four different hydroproxides formed (40) and that both the initial cleavage products (presumably free radicals) as well as some of the more stable cleavage products have reactive centers that can combine with oxygen.

"Hidden Oxidation." It has been our experience that the quality of soybean oils of unknown history and treatment is not always predictable on the basis of their peroxide values. Recent studies with oleate, linoleate, and linolenate hydroperoxides show that they decompose with heat to give both volatile cleavage products and polymeric products (41). Using liquid-liquid chromatography with methanol as the stationary phase and methanol-benzene as the mobile phase, Evans and Frankel have been able to analyze for this "hidden oxidation," *i.e.*, the polymeric products. Figure 9 shows the direct correlation between prior oxidation and polymeric products present in a given sample (42). Directions for this method have been furnished to all members of the Soybean Oil Research Conference and Soybean Research Council and are available

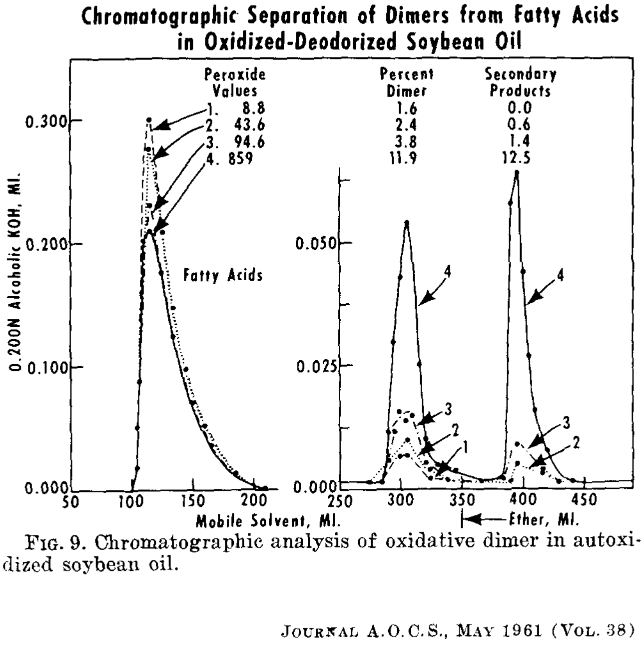


FIG. 9. Chromatographic analysis of oxidative dimer in autoxidized soybean oil.

by writing to us. The flavor stability of a given soybean oil in the absence of additives appears to be directly related to its metal, peroxide, polymeric, and tocopherol (43) contents.

Selective Hydrogenation. With renewed interest in an improved soybean oil for use both as a cooking and a salad oil, we have recently initiated studies on hydrogenation. The work includes studies on the products formed on exposure to tritium (44) and on selective hydrogenation of linolenic acid with nickel and hydrogen (45) and with hydrazine (46); it also includes a survey of commercial catalysts, development of a method for evaluating catalysts, and an investigation of hydrogenation and winterization and their effects on quality. The goal of these studies is to find means of hydrogenating soybean oil to remove linolenic acid without forming much *trans* isomer or stearic ester.

A portion of this goal was achieved in the discovery that hydrazine would reduce linolenic acid without forming *trans* isomers or moving unsaturated bonds. Figure 10 shows that hydrazine with air reduces linolenic acid XXI to give linoleic, 9,15- and 12,15-octadecadienoic, and 9-, 12-, and 15-octadecenoic acids and stearic acid without forming any *trans* bonds. Thus we have a source of new mono- and dienoic acids that was not previously available, and an antioxidant that when exposed to oxygen hydrogenates the oil.

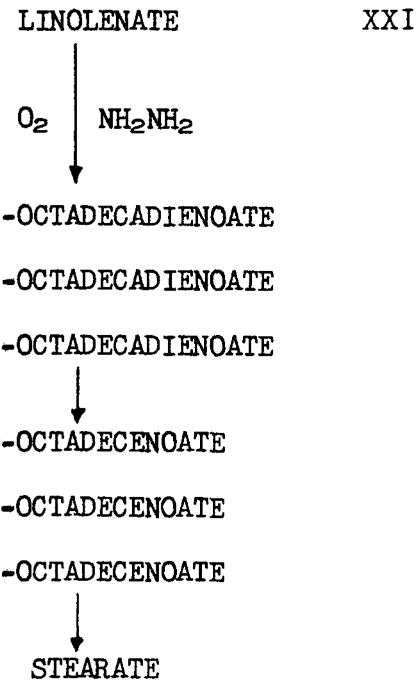


FIG. 10. Chemical reduction of linolenic acid with hydrazine.

Structure of Vegetable Oils

When H.J. Dutton undertook studies in 1955 on the distribution of the fatty acid in the glycerides of linseed oil, it was generally accepted that the structure of most vegetable oils probably approached the even distribution proposed by Hilditch (47).

Distribution of Fatty Acids in Glycerides. In our work at Peoria we have found that the fatty acids in linseed (48), soybean, safflower, and corn oil (49) approach a restricted random distribution. Our data are not complete because we have made no attempt with the pure glycerides of the mixed acids present in these oils to determine the position of the individual fatty acids.

Our recent work has added a new chapter to the structure of cocoa butter and its potential commercial synthesis. A consideration of our results on the liquid-liquid fractionation of cocoa butter led Dutton to suggest and establish that its structure was 1,3 randomly-distributed palmito-stearo-2-olein XXVII. In the synthesis outlined in Figure 11 a 1,3 random palmito-stearo-diglyceride XXVI reacts with oleyl chloride to give a product similar to cocoa butter (50). Perhaps the hardest property to achieve in a synthetic cocoa butter is mold release. Suffice to say, our synthetic product releases readily from the mold.

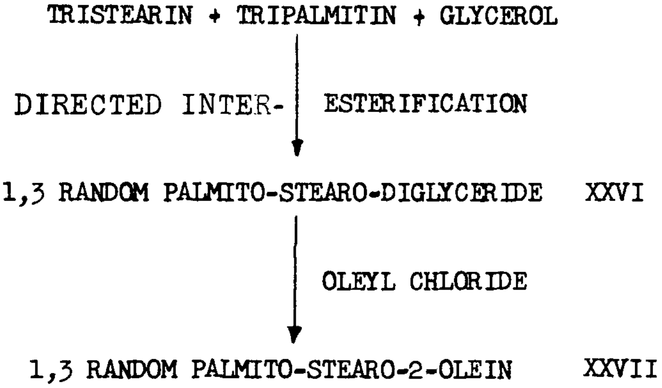


FIG. 11. Synthesis of cocoa butter.

New Vegetable Oils. In our new crops research under the direction of Ivan A. Wolff more than 2,000 samples of seeds have been surveyed and several new oils have been isolated and their structures studied. One of the unique oils is *dimorphotheca.* It contains dimorphecolic acid—a 9-hydroxy *trans,trans*-10,12-octadecadienoic acid—that readily dehydrates to a trienoic acid or adducts with maleic anhydride (51). As this work progresses, we believe that new and unique structures will be found that may lead us to new horizons in utilization of vegetable oils.

Finally I might cite the very considerable amount of work our laboratory has done or is doing on the reaction of fatty acids with maleic anhydride and maleic esters (52), emulsion paints in cooperation with the National Flaxseed Processors Association (53), countercurrent distribution (54,55) and gas chromatography of fatty esters (56). In this brief glimpse of past and present work you can see that research today is much different. The past 20 years has seen many changes in commercial and research operations on fats and oils. The next 20 years will see even more. The rate of discovery and reduction to practice appear to be accelerating. Your research as well as ours will have to be dynamic and progressive to keep pace. Two years ago in the research laboratories at Unilever at Vlaardingen, Holland, I saw 11 pieces of gas chromatographic equipment as well as a specially constructed apparatus for nuclear magnetic resonance. How many of us can even match this array of data-gathering machines for our research now? Not only do we need to push forward our use of new devices and research procedures, but we need to extend our findings to the development of new equipment, products, and processes.

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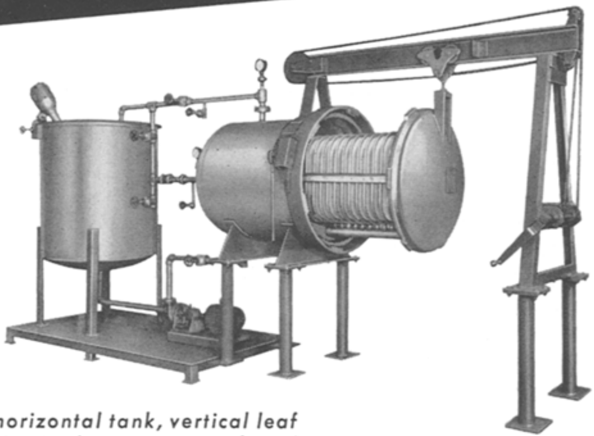

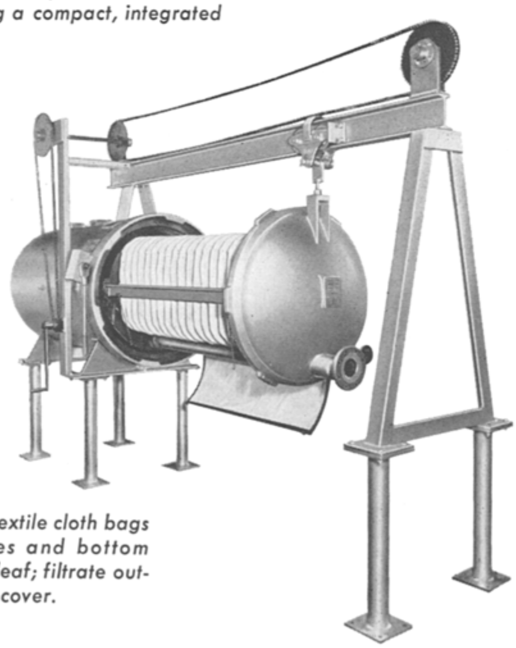

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• *A.O.C.S. Commentary*

(Continued from page 4)

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