Technical News Feature

The Role of the Northern Regional Research Center in the Development of Quality Control Procedures for Fats and Oils

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ABSTRACT

The research laboratory has played a major role in the development of quality control procedures for edible fats and oils. Historically, development of these methods can be traced in part to the need for basic information on the composition, processing and flavor stability of edible soybean oil. The Northern Regional Research Center pioneered much of this basic work. This review will address the contributions made by the Northern Center in developing quality control procedures for edible fats and oils.

INTRODUCTION

Oilseed commodity trading, quality control and forensics are just a few of the areas in which the fats and oils industry requires analytical information. Analytical methods can be grouped as follows: (a) methods for the identification of fats and oils; (b) official methods of the American Oil Chemists' Society (AOCS), Association of Official Analytical Chemists' (AOAC) and American Society for Testing Materials (ASTM); (c) methods to monitor and control levels of objectionable or toxic materials that sometimes occur in fats and oils; (d) procedures for the detection and quantitation of nontoxic materials that often have a beneficial or desirable effect in fats and oils; (e) procedures and methods to quantitate in all amounts of materials that occur in fats and oils but are mainly of academic or scientific interest. An excellent and thorough review of these methods recently appeared in A. E. Bailey's *Industrial Oil and Fat Products* and should be consulted for further information (1).

Historically, the development of analytical methods for quality control for fats and oils has evolved from producers of fats and oils, professional societies, the academic community and government-sponsored research. To discuss contributions made by all the aforementioned groups would be a formidable task, and even a complete review of government-sponsored research in fats and oils is beyond the scope of this paper. Therefore, emphasis will be placed on the contributions of the Northern Regional Research Center (NRRC) in developing quality control procedures for fats and oils.

The Regional Research Centers of the U.S. Department of Agriculture (USDA) were established by an act of Congress in 1938 and became operational by the early 1940s. Their mission was to find new uses for the large surpluses of agricultural commodities existing then. For organizational purposes, the United States was divided into four regions with headquarters and responsibilities for fats and oils research as follows: (a) Northern region, Peoria, Illinois: soybeans, flax; (b) Southern region, New Orleans, Louisiana: cottonseed, peanuts; (c) Western region, Albany, California: safflower, castor; (d) Eastern region, Philadelphia, Pennsylvania: animal and dairy fats. In 1969, a fifth Regional Laboratory, named the Richard B. Russell Research Center, was added in Athens, Georgia, with sunflowers as its prime responsibility for oilseed research.

The USDA regional laboratories have produced a long and distinguished list of scientists. The names of A. E. Bailey, K. S. Markley, D. Swern, J. C. Cowan, H. J. Dutton, R. O. Feuge, R. T. O'Connor, L. A. Goldblatt, C. D. Evans, C. R. Scholfield, F. G. Dollear, F. E. Luddy, R. W. Riemenschneider, S. F. Herb, H. S. Olcott, T. H. Applewhite, W. A. Pons Jr., I. A. Wolff and K. T. Zilch are all quite familiar to fats and oils chemists. All spent the entirety or part of their careers with the USDA Regional Centers.

SYNOPSIS OF OILSEED RESEARCH AT THE NORTHERN LABORATORY

The early years encompassing World War II were directed to alleviating shortages of rubber and plastics (2).

Shortly after World War II ended, the Northern Center undertook studies aimed toward solving the flavor problem of soybean oil. This work showed that the 7-8% linolenic acid present in soybean glyceride structure is responsible for the undesirable flavors that develop at low levels of oxidation (3,4). The beneficial effects of citric acid were demonstrated, and it was shown that metals like iron and copper need to be inactivated or chelated (5,6).

The period from 1950-1960 brought new knowledge on the glyceride structure of vegetable oils (7-11), characterization of corn and soybean phosphatides (12,13), tocopherol oxidation (14-16) and the mechanism of autoxidation (17).

During the years 1960-1970, research at the Northern Center extended into hydrogenation as a means of stabilizing soybean oil as well as into basic studies on the mechanisms of both heterogeneous and homogeneous hydrogenation (18,19). During this time, considerable method development required for an understanding of hydrogenation took place, including countercurrent fractionation of fatty acid isomers and isologs (20), double bond locations by ozonolysis (21) and argentation countercurrent fractionations of fatty acids, monoenes, dienes and trienes (22,23).

Other lines of research during the 1960s included development of instrumental methods for evaluating oil quality (24), hydrogenated winterized soybean oil (25,26) and long-term stability evaluations of edible oils (27).

More recent work at the Northern Center includes monitoring of toxic and undesirable components in fats and oils (28,29), processing of off-specification oils (30), physical refining of soybean oil (31,32), effects of antioxidants in edible oils (33), metabolic fate of isomeric fats produced during hydrogenation (34), mechanisms of fat deterioration (35,36), post-harvest handling of soybeans (37) and extraction of oilseeds with supercritical fluids (38,39,40).

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[1] The mention of firm names or trade products does not imply that they are endorsed or recommended by the U.S. Department of Agriculture over other firms or similar products not mentioned.

FINISHED OIL EVALUATION METHODS

The Northern Center was a pioneer in developing taste panel methods for evaluating the flavor stability of edible soybean oil. Today, most industrial taste panels are patterned after the methods described by Moser et al. (41,42). The tasting of paired samples under controlled conditions and statistical treatment of the data are key features of the method.

The detrimental effects of light on the flavor stability of edible oil are well known, having been reported in the early literature (43-45). Moser and coworkers took advantage of these effects and reported an accelerated method for measuring oil stability (46) by controlled exposure to fluorescent light. An apparatus for such a test was described, and deterioration occurring during light exposure was correlated with that occurring during Schall oven storage.

In 1969, under sponsorship of the American Soybean Association, H. J. Dutton, while touring European processing plants, learned that hydrogenated winterized soybean oil was considered inferior to olive and other oils that were used for frying foods in those countries. Thus, research was begun to develop organoleptic methods for assessing the quality of frying oils. In 1971, Evans et al. reported a method by which a trained odor panel could assess the quality of an edible oil after it was heated to deep-frying temperatures (47).

The handling of taste panel data by hand is a rather laborious task. To alleviate this problem, Warner et al. described a method for handling taste panel data by computer (48). Tasks that formerly required hours by hand were reduced to a matter of minutes by the use of punch cards identical to those used in voting machines.

MINOR CONSTITUENTS

The minor constituents of edible oils are important because they often are either detrimental or beneficial to stability. The Northern Center has been active in minor constituent research for many years. Prior to 1948, the soybean phosphatides were thought to consist of only phosphatidylethanolamine and phosphatidylcholine. Scholfield et al. (12) were the first to report that soy phosphatides also contain substantial amounts of phosphatidylinositol as well.

In the late 1950s and early 1960s, Frankel et al. studied the fate of soybean tocopherols during autoxidation (15) and reported that soybean oil contains more tocopherol than necessary for optimum oxidative stability. The failure of phenolic antioxidants to improve the flavor stability of soybean oil was attributed to its highly unsaturated fatty acid composition (15).

The unsaponifiable fraction of soybean oil was suggested as a cause of flavor reversion of soybean oil as early as the mid-1940s (49). However, definitive data were lacking until 1962, when Hoffmann et al. characterized the unsaponifiable fraction and showed that the hydrocarbon fraction had the most detrimental effect on stability (50).

Characterization of deodorizer distillates led to a method for isolating sterols from them (51). Evans et al. characterized the long-chain hydrocarbon present in soybean unsaponifiables (52).

More recent work on the minor constituents of vegetable oil includes a method for separation of plant pigments by high-pressure liquid chromatography (53,54).

TRACE METALS IN EDIBLE OILS

Trace metals were reported to act as prooxidants for edible fats and oils as early as the late 1930s (45). However, a clearer understanding of the magnitude of their effects required development of suitable analytical methods. The late R. T. O'Connor pioneered work on determination of trace metals in edible oils by wide-line spectroscopy (55). Melvin and Hawley (56) and Evans and coworkers (57) improved the wide-line methods and were able to show that copper and iron acted as prooxidants for soybean oil at levels of .01 and 0.1 ppm, respectively. These findings resulted in conversion from black iron to stainless steel deodorizers and in replacement of brass valves in refineries, which had significant effect on improving the quality of edible soybean oil.

By the mid-1960s, considerable research conducted by the Northern Center and others had shown that certain copper-containing catalysts were effective as selective heterogeneous catalysts for linolenate-containing edible oils (58). This work again revived interest in trace metals and led to a number of methods based on atomic absorption spectroscopy. Piccolo and O'Connor (59) and Evans and coworkers (60,61) described flame absorption in methods for determination of copper, iron and other metallic contaminants occurring in vegetable oils. Under the leadership of R. T. O'Connor and Ken Brobst, an atomic absorption subcommittee was formed to evaluate flame atomic absorption for determination of trace metals in edible oils; eventually this became a tentative method of the AOCS. By the early to mid-1970s, nonflame atomic absorption methods became available. Black (62) compared direct, ashing and nonflame absorption methods for a number of trace metals in edible oils. The results indicated that for many metals the analysis was similar regardless of technique, but zinc, cadmium, chromium lead and calcium were affected by the method used for analysis.

INSTRUMENTAL METHODS FOR PREDICTING EDIBLE OIL STABILITY

Introduction of gas-liquid chromatography in the late 1950s and early 1960s provided the fats and oils chemist with a powerful tool for the study of fat deterioration and isolation and characterization of compounds responsible for flavors in edible oils. Two of the early pioneers in this field include Professor Stephen Chang at Rutgers University and the late Cyril D. Evans of the Northern Regional Research Center.

Evans (63) apparently was the first worker to suggest that hydrocarbons were among the initial decomposition products of fat hydroperoxides, and he developed gas-solid chromatographic methods for separation of a number of hydrocarbon compounds. Activated alumina proved an effective adsorbent for the separation of alkane, alkenes, alkyne, and cyclic hydrocarbons (64-66).

Thermal decomposition of fat hydroperoxide in the injection port of a gas chromatograph yields n-pentane as a major decomposition product. Pentane, as determined by gas-solid chromatography on alumina, was shown to correlate well with the peroxide value and the flavor scores of cottonseed and soybean oil stored under Schall oven conditions (67).

By the early 1970s other groups became very active in method development for predicting edible oil quality and stability. Dupuy and workers at the Southern Center introduced the concept of separating volatile compounds from edible oils by placing a small amount on glass wool in a tube inserted into the injection port of a gas chromatograph, sweeping the volatiles onto the first part of the cooled column and finally separating the volatile compounds by temperature programming (68).

Subsequent cooperative work with the Northern Center showed that high correlations exist between the pentanal and hexanal contents and the flavor score of soybean salad oils and cottonseed and peanut oils (69).

Dupuy's (70) group later demonstrated that total volatiles from an edible oil also correlate with flavor, and similar conclusions were reached by Jackson and Giacherio (71), Williams and Applewhite (72) and Waltking and Zmachinski (73).

Thus the Northern and Southern Centers made significant contributions to development of instrumental quality control methods for edible fats and oils.

QUALITY CONTROL METHODS FOR CRUDE AND PARTIALLY PROCESSED OILS

Analytical methods that will reflect the quality of finished products from a given lot of crude oil have long been of interest to the quality control chemist. Although tests for color, free fatty acids, trace metals and refining loss are, in some cases, adequate for this purpose, other more sensitive tests are still highly researchable areas.

An early method for assessing oxidation in crude oil was developed by Holm (74) in Sweden. Her work demonstrated that carbonyl compounds react with benzidene to form a complex that can be determined spectrophotometrically. Since alpha-beta unsaturated aldehydes show the strongest reactivity with benzidene, the benzidene value is primarily a measure of this class of compound. For many years the benzidene test was used by European oil processors, and it became an IUPAC method. However, owing to its carcinogenic properties, an alternative was sought. In the early 1970s, Holm introduced an alternative reagent, *p*-anisidine (75). In our hands, the anisidine value itself, which is a measure of secondary oxidation, yielded erratic correlations with the flavor scores of edible soybean oils. However, significant correlations were found between the oxidation value (anisidine value + 2 peroxide value) and the flavor scores of finished soybean salad oils (76).

The extent of oxidation occurring prior to deodorization of an edible oil is difficult to determine because heat rapidly destroys fat hydroperoxides (77). To overcome this problem, Frankel et al. reported a chromatographic method for estimating the amount of "hidden oxidation" occurring during the processing of soybean and other edible oils (77). The decomposition products from fatty hydroperoxide were shown to be principally dimeric in nature and to contain 1 mole hydroxyl, 0.5 mole of carbonyl and 2 double bonds per mole of dimer (78). Close relationships were found between the flavor score of aged samples, their dimer content and the peroxide level of the oil prior to deodorization.

Periodically, damage occurs to the soybean crop in the field and during storage. Evans et al. reported that the phosphorus, iron and free fatty acid contents of crude soybean oil are useful undexes for estimating the extent of field or storage damage to soybeans (79).

Mounts et al. investigated the effects of post-harvest handling of soybeans on oil quality (37). Identity-preserved shipments of soybeans were sampled at origin and at destination ports. The beans were separated into wholes and splits. Crude oils extracted from each fraction were characterized for free fatty acids, iron content, peroxide value, phosphatides, nonhydratable phosphatide, color, chromatographic refining loss and fatty acid composition. Oil extracted from split beans had free fatty acids and iron contents significantly greater than those of oil from whole beans. These two deleterious qualities of crude oils were found to increase during shipment. Degumming studies showed an increase in the nonhydratable phosphatide content of extracted oil after shipping.

Subsequent cooperative studies conducted by laboratories of the University of Illinois and the Northern Center showed that export shipment produced a significant increase in the refining loss of crude soybean oil, as shown by a centrifugal refining-loss test (80).

MONITORING TOXIC AND OBJECTIONABLE MATERIAL IN FATS, OILS AND OILSEEDS

The Northern Center has been involved in development of methods for detection of toxic and objectionable materials in fats, oils and oilseeds since the late 1960s, when polycyclic aromatic hydrocarbons were reported as minor constituents in a variety of vegetable oils.

Mounts et al. (28) used a radiochemical approach to studying the effect of laboratory processing on removal of labeled benzo-(α)-pyrene from soybean oil. Bleaching and deodorization were shown to be effective in removal of these compounds.

Chlorinated dibenzo-*p*-dioxins have been determined to be the toxic substance in chick edema factor, a disease associated with contaminated fats and oils in feeds. Mounts et al. (29) studied the removal of ^{14}C-labeled 2,3,7,8-tetrachlorodibenzo-*p*-dioxin and 2,7-dichlorobenzo-*p*-dioxin from soybean oil. Deodorization and bleaching with activated carbons were effective in removing the chick edema factor from refined soybean oil.

In 1960, 100,000 young turkeys in Great Britain died after consuming rations made from moldy peanuts. Subsequent work identified the principal toxic compounds as secondary metabolites produced by the action of a mold (81). These toxic compounds, known as aflatoxins, needed to be separated, identified and quantitated. Both the Southern and Northern Center were early pioneers in developing analytical methods for the aflatoxins. Notable pioneers in this field include W. A. Pons and O. L. Shotwell of the Southern and Northern Centers, respectively. For further information on the separation and quantitation of the aflatoxins, Stubblefield et al. (82), Shotwell and Stubblefield (83) and Stubblefield (81) should be consulted.

Soybeans and other grains are subject to potential contamination from toxic weed seed during harvest, transportation and storage. The more common foreign seeds include those from jimson weed, cocklebur, morning glory, castor, poke weed and crotolaria (84). Of these seeds, jimson weed is, without a doubt, the most common toxic seed. Occasionally, herbicides are not effective in controlling this weed and, in 1973, sorghum exported to India was badly contaminated with jimson weed seed; the jimson weed seeds are about the same size and color as sorghum, and thus cannot be separated during harvest or cleaning. That incident prompted investigations concerning the analysis, distribution and processing of jimson weed seed alkaloids. Work at the Northern Regional Research Center indicates that jimson weeds typically contain ca. 0.29% and 0.05% of atropine and scopolamine, respectively (84).

To determine distribution of alkaloids, extraction of a mixture of soybeans and jimson weeds was carried out with hexane to yield crude oil and meal. Analysis of both fractions showed that virtually all of the alkaloids resided in the meal (85). Caustic refining effectively removed atropine from a sample of crude oil spiked with this alkaloid.

QUALITY CONTROL METHODS FOR THE ANALYSIS AND IDENTIFICATION OF FATS AND OILS

Prior to about 1955, vegetable oils were thought to conform to the even theory of glyceride structure, i.e., fatty acids found to be spread as evenly as possible over the glycerol moiety. However, at about this time, H. J. Dutton published the first of a series of papers on the glyceride structure of vegetable oils by countercurrent distribution (7).

These studies provided evidence that the fatty acids of linseed, corn and soybean oil approach a restricted random distribution (7-11). The pioneering work of Dutton and coworkers, coupled with the discovery that pancreatic lipase cleaves fatty acids from the 1,3 position of glycerol and the introduction of gas chromatography, stimulated a very active period in glyceride structure research. By 1962, the work of Mattson and Volpenhein (86), Coleman (87) and Vanderwal (88) provided strong evidence that nearly all vegetable oils conform to a restricted random structure known as the 1,3 random-2 random theory of glyceride distribution.

Unequivocal proof of this theory was demonstrated by the work of Evans et al. (89), who isolated pure natural triglyceride isomers from linseed, soybean and safflower oils by countercurrent distribution. Lipase hydrolysis studies showed that the structures of the isolated glycerides closely matched those predicted by the 1,3 random-2 random theory advanced by Coleman and Vanderwal.

The work of Evans also led to a method whereby the glyceride structures of many vegetable oils can be estimated from their fatty acid composition (90).

MASS SPECTROMETRY, GAS AND HIGH-PRESSURE LIQUID CHROMATOGRAPHY

To say that the discovery of gas chromatography by James and Martin in 1956 provided the fats and oils chemist with a powerful tool for studying the composition of fats and oils would be an understatement.

By 1957, the Northern Center had published a method for separation of fatty acid oxidation products by gas-liquid chromatography (91).

In the 1960s the Northern Center began an intensive search for new industrial oils, and by the end of the decade had characterized hundreds of new oils by gas-liquid chromatography and other ancillary techniques (92-99).

Early NRRC contributions to the development of gas chromatography include identification constants for mono- and decarboxylic methyl esters (100), identification of gas chromatographic peaks (101) and a graphic aid for interpretation of gas chromatograms (102). Hoffmann showed that significant improvements in detector sensitivity can be achieved by cryostatic operation of a thermal conductivity (103) cell and through hyperoxygenation of a flame ionization detector (104). The thermal conductivity of carrier gases was shown to affect the sensitivity of a flame ionization detector (105), whereas other investigations showed that the choice of carrier gas has an effect on retention time and column efficiency in gas-solid chromatographic separations on activated alumina (106).

High-pressure liquid chromatography (HPLC) is a powerful tool for the analysis and characterization of fats, oils and their minor constituents. Notable NRRC contributions in this area include the HPLC separation of triglycerides (107), reverse-phase HPLC separation of triglycerides (108) and HPLC quantitation of free fatty acids, mono-, di- and triglycerides with an infrared detector (109). The high-performance liquid chromatography of triglycerides has been reviewed in detail by Plattner (110). Scholfield has described argentation in HPLC separation of fatty methyl esters (111). An HPLC method for separation of vitamin E isomers was described by Cavins and Inglett (112).

The Northern Center has been active in mass spectrometry and mass spectrometry/mass spectrometry research for many years. These techniques have been applied to the analysis in characterization of fatty acid triglycerides (113,114), alkaloids (115), phospholipids (116), waxes and wax esters (117-119), deuterated fats (120) and mycotoxins (121). A method for determining the headspace gases in bottled vegetable oils by mass spectrometry has been described by Evans and Selke (122).

ANALYSIS AND METABOLISM OF ISOMERIC FATS

Method development for the analysis of isomeric fats has been under study at NRRC since the early 1960s. Early work in this area was directed towards a more thorough understanding of the mechanism of catalytic hydrogenation of fats. More recent efforts have shifted toward understanding the metabolic fate of isomeric fats in human nutrition. A detailed review of these areas is unnecessary, because they are thoroughly discussed in recent AOCS monographs (123,124).

The detection of adulteration of fats and oils becomes important in both domestic and international trading channels. The Northern Center has been active in methods development for authentication of commercial fats and oils. Spencer et al. (125) investigated the fatty acid composition of 200 samples of commercial fats and oils; they compared them to the standard ranges specified by the Food and Agriculture Organization/World Organization in the Codex alternative committee and fats and oils. Only 6 samples fell notably outside the standard ranges and, on this basis, the U.S. Codex delegation offered a method for acceptance of a sample as labeled. Spencer et al. have also described a simple graphic procedure for the identification of fats and oils within the Codex alimentarius guidelines (126).

Space does not permit a thorough and detailed review of all contributions made by the Northern Center over a period of 40-plus years of research. Furthermore, the research carried out by the Horticultural and Special Crops group (formerly Industrial Crops) at NRRC has not been given adequate space; as mentioned previously, this group has characterized thousands of seed oils, Many new fatty acids and minor constituents of seed oils have been discovered and characterized. Further information on this research can be found in numerous reviews authored by NRRC scientists (127-129).

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