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ANTIOXIDANTS

New Developments for Food Use

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The use of antioxidants in fats and in foods containing fats has increased greatly during the past 10 years. In addition to preventing loss from impairment of palatability, antioxidants conserve the nutritive value of foods, and thus are making an important contribution to better nutrition and to conservation of our food supply. Several substances isolated from natural sources have been found effective in increasing the keeping time of fats as measured by the active oxygen method, but none of them have the important property of "carry-through." Two synthetic antioxidants, butylated hydroxyanisole and 2,6-*tert*-di-butyl *p*-cresol, have excellent carry-through properties. Butylated hydroxyanisole is now used extensively in lard and other edible fats, breakfast cereals, fried corn crisps, potato chips, and nuts, and to treat paraffin-coated wrappers and paper wrappers used in food containers. It is estimated that currently at least 50% of the federally inspected lard produced in this country is stabilized with this antioxidant.

THE USE OF ANTIOXIDANTS to retard rancidity in food fats and foods containing fats is making an important contribution to better nutrition and conservation of our food supply. The development of rancidity in foods lowers the palatability and impairs the nutritive value. Precise data are not available on the amount of food lost owing to rancidity, but it is known to be a considerable amount and experience shows that the use of antioxidants has materially reduced this loss. Rancidity in foods impairs the nutritive value by reducing the content of carotene, various fat-soluble vitamins, and essential fatty acids. There is evidence to show that destruction of these nutrients begins before rancidity can be detected organoleptically. Thus in addition to prevention of loss through impairment of palatability, the use of antioxidants is important in conserving the nutritive value of foods. Rancidity is not confined to foods of high fat content. It may occur in cereals and other foods of relatively low fat content, and under conditions of storage which retard or prevent microbial spoilage, such as refrigeration, dehydration, and salting.

The use of antioxidants in foods, cosmetics, rubber, gasoline and other petroleum products, vitamins, insecticides, and dyes has increased greatly during the past 10 years. During this period about 600 United States patents on antioxidants have been issued.

In 1947 Lundberg (*38*) published a comprehensive review of the antioxidants then in use and proposed for use in fats and foods. Since that time a number of other substances have been proposed and put in use. This paper reviews briefly some of the more recent developments in antioxidants in foods.

Antioxidants from Natural Sources

A number of substances isolated from natural sources have been found to have antioxidant properties and have been proposed for commercial use. Some of these products could be made available in large quantities at low prices. Others probably could not be produced economically. Although some of these naturally occurring substances are effective in increasing the keeping time of fats as measured by the active oxygen method, none have been shown to have the important property of carry-through. [The term "carry-through" is used to denote the effect of the antioxidant in retarding development of rancidity in foods made with fat, as, for example, pastry, crackers, and potato chips.]

Norconidendron was reported by Fisher *et al.* (*18*) in 1947 to be an effective antioxidant for certain vegetable oils. This substance was prepared from conidendron isolated from sulfite waste liquor of the western hemlock. Norconidendron has since been resolved into two isomeric substances, α- and β-conidendrol. Mack and Bickford (*40*) found these substances effective as antioxidants for fats, oils, and candies, as measured by the active oxygen method. No data were reported to show whether the effectiveness carries through into the foods made with the fats. Toxicity studies are being conducted on the conidendrols.

Dihydroquercetin, isolated from the bark of the Douglas fir and Jeffrey pine, was reported by Kurth and Chan (*36*) to be effective as an antioxidant for lard, butter oil, and cottonseed oil. It imparts no color, odor, or taste to fats and is believed to be nontoxic. The authors have found in their laboratories that it is about as effective as propyl gallate in increasing the AOM stability of lard. It does not have the important property of carry-through into the foods made with lard to protect them from rancidity.

Budowski (*6*) found that sesamol, isolated from sesame oil, improved the stability of lard, peanut oil, cottonseed oil, and sesame oil. No data were reported on carry-through properties.

Chipault *et al.* (*8*) reported that 32 ground spices enhanced the stability of lard. Rosemary and sage were particularly potent as antioxidants. The antioxidants contained in these natural spices were not effectively carried through into the foods made with lard.

An extract from the fruit of Osage orange has been suggested for use as an antioxidant. Quackenbush *et al.* (*50*) and Clopton (*10*) showed that an extract containing chiefly pomiferin with some osajin, both isoflavones, and some resinous materials increased the active oxygen method stability of lard. Pomiferin is the most active ingredient of the extract. The extract is believed to be nontoxic and in useful concentrations imparts no noticeable flavor or odor. No data are given regarding the effect on color of the lard and the carry-through properties.

Synthetic Antioxidants

Numerous antioxidants have been synthesized for use in fats and oils, fatty foods, carotene, feeds, rubber, and gasoline and other petroleum products. Most of these substances are substituted phenols and polyphenols or aromatic amines. Many of the compounds are not suitable for use in edible fats or foods because they are toxic or impart an objectionable color, odor, or flavor to the foods. Most of the compounds found effective in carry-through properties belong to the class of hindered or partially hindered phenols. Outstanding among these are butylated hydroxyanisole and 2,6-di-*tert*-butyl *p*-cresol.

Butylated hydroxyanisole, commonly known as BHA, was proposed for use in animal fats in 1948 by the authors' laboratories after extensive studies (*4, 13, 14, 33–35*). The commercial product as used today consists chiefly of two isomers, 3-*tert*-butyl-4-hydroxyanisole and 2-*tert*-butyl-4-hydroxyanisole. The 3-*tert*-butyl-4-hydroxyanisole was synthesized by Rosenwald and Chenicek (*57*) and proposed for use in gasoline.

The authors found, in 1945, that butylated hydroxyanisole is unique among the many compounds tested. It is very much more effective than the other compounds tested in carry-through properties. The foods prepared with butylated hydroxyanisole stabilized lard —for example, pastry, crackers, and potato chips—show a marked increase in resistance to rancidity. When butylated hydroxyanisole is used in combination with propyl gallate and citric acid, the AOM values are increased greatly. The antioxidant is effective in low concentrations and imparts no objectionable odor, flavor, or color to the lard or foods made from the lard. Butylated hydroxyanisole and combinations containing butylated hydroxyanisole (*14*) were found effective in retarding rancidity in corn chips, nuts, dry dog food, chicken fat, and wrapping papers. After extensive toxicity studies in the authors' laboratories and in the Department of Physiology of the University of Chicago, which showed that butylated hydroxyanisole was safe for use in foods, the Federal Meat Inspection Service, in 1948 (*61*), with the approval of the Federal Food, Drug and Cosmetic Administration, issued regulations permitting the use of the antioxidant in edible animal fats.

The compound, 2,6-di-*tert*-butyl *p*-cresol was developed as an antioxidant for gasoline and petroleum products (*56*). The authors found that the product used commercially in petroleum products is effective as an antioxidant for animal fats. When the commercia product is used in lard, discolorations, off-flavors, and odors develop in the foods made with the stabilized fats, but these can be overcome when the preparation is highly purified. Extensive studies have shown that 2,6-di-*tert*-buty *p*-cresol is slightly more effective than butylated hydroxyanisole in increasing the AOM value but is not as effective as butylated hydroxyanisole in carry-through. Combinations of butylated hydroxyanisole and 2,6-di-*tert*-butyl *p*-cresol exhibit marked synergism in AOM values. The synergistic effect carries over into the stability of the heated fat. A lard of 11 AOM hours treated with 0.02% of 2,6-di-*tert*-butyl *p*-cresol had an AOM value of 64.75 hours. The same lard treated with 0.02% of butylated hydroxyanisole had an AOM value of 54.5 hours. When treated with 0.01% butylated hydroxyanisole and 0.01% of 2,6-di-*tert*-butyl *p*-cresol the lard had an AOM value of 101 hours. The value is 36.25 and 46.5 hours higher, respectively, than when an equivalent amount of either of the single antioxidants is used. 2,6-Di-*tert*-butyl *p*-cresol has not yet been approved for use in foods. Toxicity studies are nearing completion and there is every reason to believe that the compound will be approved for use in the near future.

The authors found the rubber and gasoline antioxidant 2,2′-methylenebis-4-methyl-6-*tert*-butylphenol to be more effective in increasing the AOM stability of lard than butylated hydroxyanisole or 2,6-di-*tert*-butyl *p*-cresol, but it is not as effective in carry-through. Preliminary LD_{50} studies showed that the compound was low in acute toxicity, but the Federal Food and Drug Administration (*24*) has shown that it possesses certain harmful physiological activities which would preclude its use in foods.

Acid Synergists as Metal Scavengers

The role of acid synergists in increasing the effectiveness of antioxidants may be due in part to their chelating action with trace metal pro-oxidants. The use of metal scavengers has been found effective in delaying reversion in soybean oil and products made from it. Dutton *et al.* (*16*) reported the addition of either citric acid or sorbitol during deodorization effective in improving oxidative and flavor stability of soybean oil. Neal, Gooding, and Valteich (*21, 22, 46, 47, 62, 63*) found the mono- and dialkyl or mono- and dialkylene esters of citric acid effective in stabilization of nut meats, cheese, margarine, and shortenings. Watts (*65*) showed that a group of polyphosphates are effective as synergists in water-fat systems. Clausen *et al.* (*9*) and Lundberg (*39*) found that certain amino acids act as synergists with some of the phenolic antioxidants. Recently Evans *et al.* (*17*) found phytic acid an effective agent for inactivating trace metals. Schwab *et al.* (*53*) reported that nitrogen coordination compounds are effective as metal deactivators in edible oils. Cowan and Evans (*11*) found starch phosphates and Schwab and Dutton (*54*) found β-stearoxy and β-palmitoxy tricarballylic acids effective metal inactivators for fats and oils.

Food Uses of Antioxidants

The use of butylated hydroxyanisole and combinations of antioxidants containing butylated hydroxyanisole has made it possible to prepare shortenings from animal fats with stabilities comparable to those of hydrogenated vegetable shortenings. Because of the uniquely high degree of carry-through possessed by butylated hydroxyanisole, the food products prepared with the stabilized animal fats have excellent resistance to rancidity. It is estimated that currently at least 50% of the federally inspected lard produced in this country is stabilized with butylated hydroxyanisole. The development of improved methods of processing and the use of an antioxidant with good carry-through properties have made it possible to produce shortening of excellent quality from animal fats. In 1946, 20,000,000 pounds of lard were used in this country in the manufacture of shortenings and in 1952, 218,000,000 pounds.

Lard is used extensively in manufacture of soda crackers and other baked products. Micka (*43*) recently stated that, if lard stabilized with butylated hydroxyanisole to an AOM value of 30 to 50 hours is used in crackers, the crackers remain free from rancidity even in the summer season. Goss (*23*) reported that, when antiodxiants are used in good fats, there is no problem of rancidity in either grocery or bakery-prepared mixes. Antioxidants of the type containing butylated hydroxyanisole, propyl gallate, and citric acid are used extensively for preserving freshness of breakfast cereals, deep-fat-fried corn crisps, and potato chips (*14, 42*).

Klose *et al.* (*31*) found butylated hydroxyanisole, propyl gallate, and combinations of these antioxidants effective in reducing deterioration of fat during frozen storage when applied in various carriers to the natural and cut surfaces of eviscerated turkeys. Lineweaver *et al.* (*37*) reported that the application of butylated hydroxyanisole or combinations of butylated hydroxyanisole, propyl gallate, and citric acid during the cooking of turkey retards the development of rancidity during cooking and subsequent frozen storage. These antioxidants are now being used in precooked frozen beef pies and chicken pot pies.

Antioxidants are being used to prevent rancidity in nuts and foods made with nuts (*14*). Godkin *et al.* (*20*) found that pecans were protected by a 40% sucrose sirup, but that nordihydroguaiaretic acid failed to increase the effectiveness. Cruess and Armstrong (*12*) reported that walnut kernels treated with nordihydroguaiaretic acid were more stable, but they found as Godkin did that the nordihydroguaiaretic acid imparted a bitter taste to the nuts. Tannic and digallic acids were also found effective. Magoffin and Bentz (*42*) found that the use of butylated hydroxyanisole in the cooking fat and on the salt increased the shelf life of potato chips. Cecil and Woodroof (*7*) found that the use of butylated hydroxyanisole in the cooking fat and/or on the salt increased the shelf life of pecans, peanuts, and peanut butter. The outstanding effect of the use of butylated hydroxyanisole in peanut butter was the increased time that the product retained the natural fresh aroma and flavor. Miers and Owens (*44*) report delay in rancidity in shelled roasted nuts when the nuts are coated with a film of calcium pectinate containing an alkyl hydroxyanisole and ethylenediaminetetraacetic acid or its salts. Hall (*25*) and Sair and Hall (*26, 52*) reported increase in shelf life when an antioxidant-coated salt was used on fat-fried potato chips and nuts.

Van Blaricom and Martin (*64*) found that the loss of red color in dried and ground red cayenne pepper can be retarded by addition of antioxidants. Preparations containing butylated hydroxyanisole, propyl gallate, and citric acid; corn oil, lecithin, and propyl gallate; and ground sesame seed were effective. Flores and Morse (*19*) found nordihydroguaiaretic acid and propyl gallate effective in protecting orange oil from deterioration. Kenyon and Proctor (*30*) found nordihydroguaiaretic acid and tocopherol more effective than propyl gallate in citrus oils.

Extensive studies in this country and in a number of foreign countries have shown favorable results of the use of nordihydroguaiaretic acid, the gallates, and butylated hydroxyanisole in delaying rancidity in dairy products. Because of food regulations and laws, antioxidants are not used commercially in dairy products in this country.

Ascorbic acid is used successfully commercially in a number of foods. It has been found useful to prevent surface darkening and development of off-flavors in canned fruit (*3*), in carrots (*67*), frozen fruit, dried fruit, and fruit uices (*57, 66*). It is more effective than ethyl gallate or certain enediols in protecting salmon steaks against rancidity and off-flavors (*58, 59*). Ascorbic acid has been proposed to improve the color of cured meats (*5, 28*).

Antioxidants have been used experimentally in the curing of bacon. Komarik and Hall (*32*) described a curing process in which the pork bellies are soaked in a bath containing antioxidant for 8 to 12 hours at 38° to 42° F. Nordihydroguaiaretic acid, ascorbic acid, and combinations of butylated hydroxyanisole, propyl gallate, and citric acid were used. Hanley *et al.* (*27*) studied the effect on stability of bacon when antioxidants were injected into the belly or applied as a surface coating. Hydroquinone, butylated hydroxyanisole, propyl gallate, and citric acid were used separately and in different combinations. Dipped coating with lard containing 5 and 10% of butylated hydroxyanisole gave marked increase in stability of the bacon.

Antioxidants have been used successfully commercially for a number of years to treat paraffin-coated wrappers and paper wrappers (*14*). The oil- and fat-soluble antioxidants butylated hydroxyanisole and 2,6-di-*tert*-butyl *p*-cresol are effective in retarding rancidity of the waxes used to coat the paper. Butylated hydroxyanisole currently is used extensively for this purpose. The use of a fat-soluble antioxidant in the paper bag or wrapper which comes in contact with the food serves to delay rancidity in the film of fat from the food which spreads on to the container or wrapper. In many instances, when antioxidants are not used this film of fat is responsible for the first development of off-odors of the product. This is of special significance in transparent packages of nuts, potato chips, corn chips, and similar products. Maclay and Owen (*41*) described a pectinate film for packaging candied fruits, meats, and frozen foods in which an antioxidant is dispersed. Mitchell *et al.* (*45*) incorporated the antioxidant gum guaiac in an emulsion coating for frozen meats.

Antioxidants are used to stabilize animal fats that are added to feeds. Neumer and Dugan (*48*) showed that butylated hydroxyanisole or mixtures containing it and also 2,6-di-*tert*-butyl *p*-cresol are effective in delaying rancidity in dry dog food to which stabilized animal fat is added. During the past year there has been a rapid increase in the use of stabilized animal fats in mixed feeds and in dehydrated alfalfa meal. Among the benefits claimed are: overcoming of dustiness, improvement of stability and utilization of carotene and fat-soluble vitamins, increased efficiency of feed utilization, and improved palatability (*1*). The authors believe that the animal fats should be stabilized to a minimum of 20 hours AOM with butylated hydroxyanisole or a combination containing butylated hydroxyanisole.

Thompson (*60*) showed that several antioxidants are effective in stabilizing the carotene in dehydrated alfalfa. He found 6-ethoxy-2,2,4-trimethyl-1,2-dihydroquinoline the most effective of those tested. Several large manufacturers now are adding to dehydrated alfalfa from 1 to 2% of animal fat stabilized with butylated hydroxyanisole, propyl gallate, and citric acid. Schweigert and Siedler (*55*) found that animal fat when stabilized with a combination of butylated hydroxyanisole, propyl gallate, and citric acid and added to dry dog food is effective in retarding vitamin A losses during storage.

Some studies have been carried out to determine whether antioxidants would be of value in preventing or retarding the development of off-flavors and odors in foods sterilized with beta or gamma radiation. It has been shown that foods can be sterilized with these radiations, but off-flavors and odors develop in many foods which render the sterile products unpalatable. At least some of the off-flavors and odors have been shown to be due to oxidative reactions. Dunn *et al.* (*15*) found an increase in peroxide value of butter and olive oil subjected to gamma radiation. Astrack *et al.* (*2*) reported that the radiation-initiated reaction mechanism is complex and includes a variety of oxidative changes. Proctor and Goldblith (*49*) found that the ascorbic acids and their salts acting as free radical acceptors were effective in retarding development of off-flavor in chopped luncheon meat subjected to cathode rays. They found also that crystalline pepsin is protected and the hemolysis of red blood cells retarded by the same treatment. Huber, Brasch, and Waly (*29*) also reported that free radical acceptors retarded development of off-flavors. Spices and press juices from such organs as liver, brain, and spleen were found effective. As most of the organoleptic changes occurring under the effect of radiations are apparently due to free radicals, it appears that antioxidants may be useful in preventing these changes and that further studies should be made.

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