

Oxidation and Antioxidants in Fat and Oil Processing

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ABSTRACT

The roles of oxidation and antioxidants in the various major processing steps in producing edible fats and oils are reviewed, including the basic nature of the oxidation problem, the function of phenolic antioxidants in overcoming it, and a listing of antioxidants currently available. In addition, some important considerations in achieving best results with antioxidants, whether added during processing or to the finished fat or oil, are emphasized.

INTRODUCTION

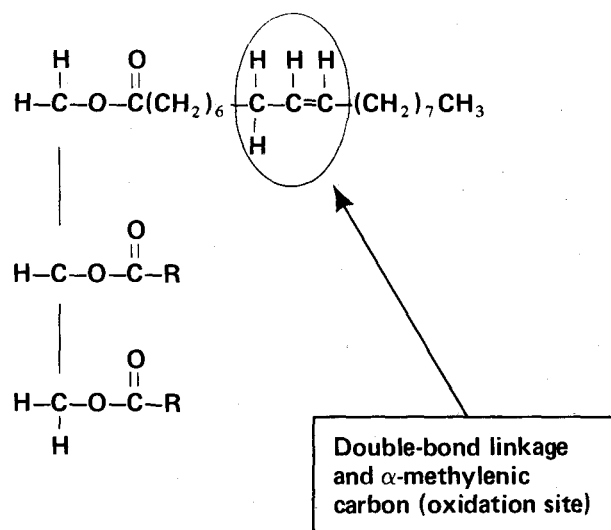
Previous presentations in this short course on Processing and Quality Control of Edible Fats and Oils have dealt with the major processes or manufacturing techniques used to produce from animal and vegetable raw materials the wide variety of high quality, useful edible fat and oil products available on the market today. Common to those manufacturing processes and also to the final fats and oils that result from them is oxidation, which generally is looked upon as being a principal route of deterioration and potential economic loss. Oxidation is found to interfere seriously with efficiency of the processing steps; it can result in organoleptic rancidity in the finished products making them unacceptable to consumers; and it can cause other degrading effects such as discoloration, vitamin destruction, nutritional losses, and polymerization.

With an adequate understanding of how this oxidative degradation actually takes place in fats and oils and with recognition of where and under what particular circumstances it most likely will occur throughout the chain of production processes, the producer can most readily take appropriate steps to eliminate, or at least minimize it as a major obstacle in the way of his producing high quality fats and oils. This may be accomplished largely through certain techniques and precautions to be incorporated in the manufacturing processes and also through the proper and efficient use, either in-processing or in the finished fats or

oils, of antioxidant substances.

OXIDATION OF FATS AND OILS

Details of how oxidation of fats and oils takes place (1,2,3,4) show that essentially it is a degradation process which occurs at the double-bond (unsaturation) sites (Figure 1) in glyceride molecules – the building blocks of edible fats and oils. Of course, the more unsaturation (i.e., the more double bonds in the molecules), as determined by the types of fatty acids making up the triglyceride structure, the more susceptible fats and oils are to oxidative breakdown. This oxidation in fats and oils may be described as in Figure 2. This demonstrates that fat and oil oxidation takes place in a series of steps and indicates why it commonly is referred to as a "free-radical" mechanism. The initiating step is the formation of a fatty free radical when hydrogen (actually a proton) departs from the α -methylene carbon in the unsaturated fatty acid group of the fat molecule (RH). The resultant free radical (R \cdot) becomes very susceptible to attack by atmospheric oxygen to form an unstable peroxide free radical (ROO \cdot). This is additional reason for the oxidation to be described as a "free-radical" mechanism. Most importantly, these free radicals themselves serve as strong initiators and promoters (catalysts) of further oxidation, hence oxidative breakdown of fats and oils becomes a self-perpetuating (autocatalytic) process giving rise to a chain reaction as may be shown in Figure 3. This shows how oxidation (as measured by formation of peroxides) progresses in animal fat (lard) or vegetable oil (cottonseed) during relatively few hours of exposure to the aeration and heating conditions of the AOM test (5) – a standardized method for fat and oil stability evaluation. Here, it is important to note the periods of time (generally referred to as induction periods) during which there is essentially no oxidation – little or no peroxide formation – and leading up to the points in time where autoxidation, or the chain reaction, commences. This is marked by the abrupt increase in the rate of oxidation, in this case at about 30 hr for the cottonseed oil and slightly over 60 hr for the lard. Without antioxidant treatments, the induction periods for both these would be much shorter. For purposes of the AOM test, the peroxide levels of 20 meq. for lard and 70 meq. for cottonseed oil are arbitrarily selected as the end points. Thus, the AOM stability values would be 73 and 50 hr for the lard and the cottonseed oil, respectively. In the final or terminating stage of glyceride oxidation, following the induction period, hydroperoxides that form in ever increasing



Where:

R = Fatty Acid Group

FIG. 1. Typical unsaturated triglyceride molecule with double-bond (oxidation site).

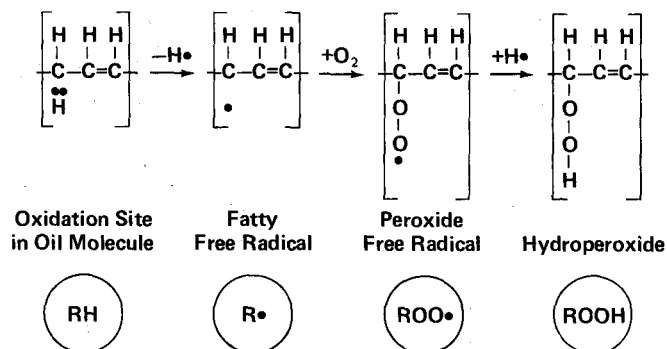


FIG. 2. Free radical oxidation of vegetable oil.

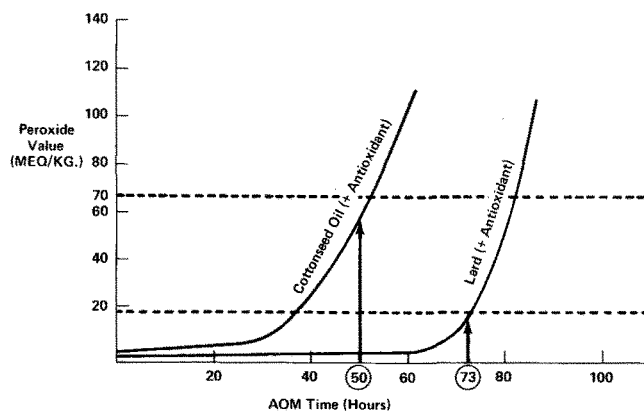


FIG. 3. Development of peroxides in lard and cottonseed oil during AOM evaluation.

TABLE I

Antioxidants Approved for Use in Edible Fats and Oils

BHA
BHT
Propyl gallate
TBHQ
Tocopherols
Lecithin
Gum or resin guaiac
THBP (trihydroxybutyphenone)
4-Hydroxymethyl-2,6-ditert. butylphenol
Thiodipropionic acid and dilauryl ester
Glycine

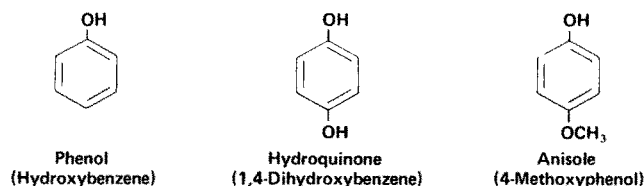


FIG. 4. Typical phenolic structures.

quantity split or decompose into short chain aromatic organic compounds (mainly aldehydes, ketones, alcohols, and acids), which actually cause the rancidity condition that ultimately destroys acceptability and usefulness of fats and oils.

An understanding of these few basic concepts about how oxidation takes place in fats and oils is fundamental to design, operation, and maintenance of optimum manufacturing processes for edible fat and oil products. There are, however, other factors, either inherent in the processing stages or introduced from outside sources, that can initiate or strongly catalyze oxidation. These factors must be recognized and eliminated to the maximum possible extent in the interest of minimizing oxidation problems in fats and oils and to insure production of top quality, stable finished products. Major factors (6) of this type are:

Enzymes - organic catalysts (lipases and lipoxygenases) naturally present in all animal and plant tissues. Upon activation by certain conditions of heat and moisture, they strongly catalyze hydrolytic and oxidative decomposition of fats and oils.

Metals - in just trace amounts are recognized as the predominant prooxidant materials encountered in commercial fats and oils (7). It is estimated that copper or iron at concentrations of less than 1 ppm can cause very serious reduction of fat or oil stability. This problem is

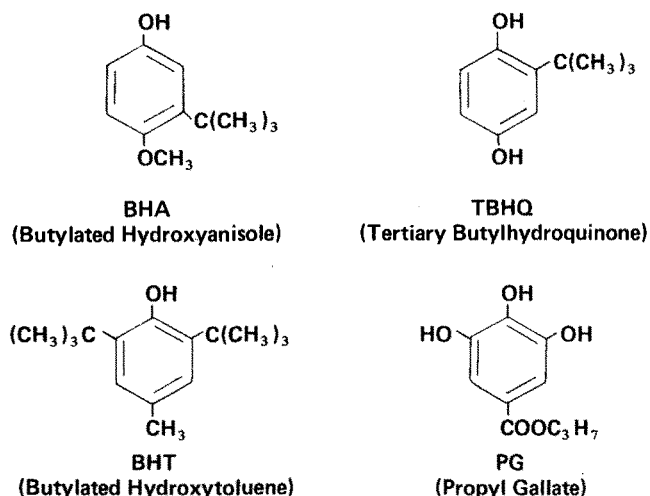


FIG. 5. Molecular structures of antioxidants most commonly used in edible fats and oils.

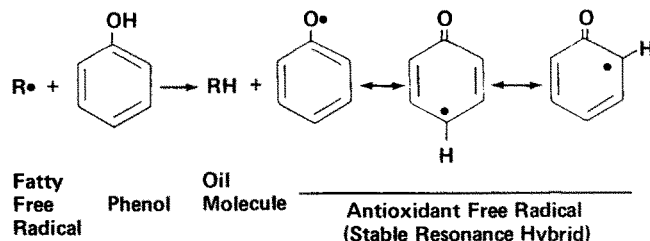


FIG. 6. Phenolic antioxidant mechanism in vegetable oil.

magnified by free fatty acids which act to solubilize metals in fat or oil.

Heat - greatly accelerates oxidation, especially at higher temperatures (above 60 C) where it has been estimated (7) that for ca. 15 C increase in temperature the rate of the oxidation reaction doubles.

Light - especially ultraviolet or near ultraviolet, has a strong accelerating effect on fat and oil oxidation.

Aeration - brings oxygen into intimate contact with the fat or oil and, at the elevated temperatures common to the various production processes, strongly accelerates oxidation.

ANTIOXIDANT MECHANISM (4) AND ANTIOXIDANTS

Since, as described earlier, the "chain reaction" of glyceride autoxidation is initiated and propagated by the formation of free radicals, it follows that removal or inactivation of the fatty or peroxide free radicals should terminate, or at least interrupt, fat oxidation in its early stages and thus delay breakdown into the final end products that are responsible for rancidity. That is the function of the substances in Table I which are cleared under government regulations for use as antioxidants in food fats and oils. The majority of requirements for added antioxidant protection in fats and oils are being filled with the first four compounds in the listing. Mostly, antioxidant addition is to finished fats and oil, but there are some instances where they may be used beneficially as "in-process" inhibitors. Either way, however, the effectiveness of these substances as antioxidants for glycerides stems from the phenolic configuration (Figure 4) within the molecular structures of the more commonly used antioxidant compounds (Figure 5), which often are referred to as phenolic antioxidants.

Figure 6 depicts how the phenolic substance, func-

tioning as a free radical acceptor, can terminate fat or oil oxidation at the initiation stage. As illustrated, the antioxidant free radical that results is stable and, most importantly, it will not initiate or propagate further oxidation of the glyceride. In view of this role of phenolic antioxidants in fats and oil, it becomes apparent that maximum efficiency is to be achieved only if they are completely mixed or dissolved in the fat or oil and if they are added soon enough, either in a processing stage or in the finished oil, to interrupt the free radical oxidation mechanism.

OXIDATION AND ANTIOXIDANTS IN FAT AND OIL PROCESSES

Now, with that brief background on how oxidation takes place in fats and oils, on the major factors that may influence oxidation in fat and oil processes, and on the types of antioxidants available and how they function, let's examine some of the important roles of oxidation and antioxidants in the various fat and oil production processes.

Extraction, the initial processing step, may be accomplished by rendering, expression, or solvent techniques designed to obtain crude fat or oil from a wide variety of animal and vegetable sources in the best condition, free as possible from nonglyceride impurities. At this early point in production, there are two most important considerations. First, present in all animal and plant tissues are enzymes (mainly lipase and lipoxygenase) which initially aid in the formation of fats, proteins, and carbohydrates in living tissues, but upon death of the animal or plant they reverse their role and cause decomposition of those materials, including the fats, they originally helped to form. Such enzyme-catalyzed hydrolytic and oxidative decomposition has serious effects (8) on refining yields and on the quality of finished fats and oils and must be prevented. This is accomplished by inactivating the enzymes, usually by applying adequate heat (50-70 C) to the raw materials early in the extraction process. Natural antioxidants (tocopherols) in plant tissues do not appear effective in counteracting the enzyme-induced oxidation and, in fact, they, too, may be destroyed in the process, thus contributing to lowered quality of the finished oil.

Secondly, much attention has been given in recent years (8, 9, 10) to the harmful effects of using damaged raw materials such as abused palm fruits and weather-damaged soybeans on ability to produce high quality, stable oils. Use of such damaged oil-bearing materials can result in serious refinery losses as well as finished oils with lowered inherent oxidative stability, poor response to added antioxidants, and impaired color.

In the past few years, considerable work has been done (11, 12, 13) on adding antioxidants to various types of crude oils to inhibit oxidation which can take place during handling and storage of the oil, ultimately resulting in significant impairment in quality of the finished oil. Results reported from this work indicate that not only do added antioxidants such as TBHQ suppress oxidation during storage of the crude oil, but the refined, bleached, and deodorized oils produced from the antioxidant-protected crude oils have somewhat higher initial oxidative stability and are observed to respond better to treatment with additional antioxidants.

This sort of oxidation problem with crude vegetable oils has come to the forefront most recently with palm oil which, because of its relatively high melting point, commonly is subjected to heating for long periods to keep it molten during shipment and storage. Under these adverse time-temperature conditions, and particularly with the relatively high levels of prooxidant trace metals which may be present, crude palm oil is susceptible to oxidation, especially related to its high carotene content, which can

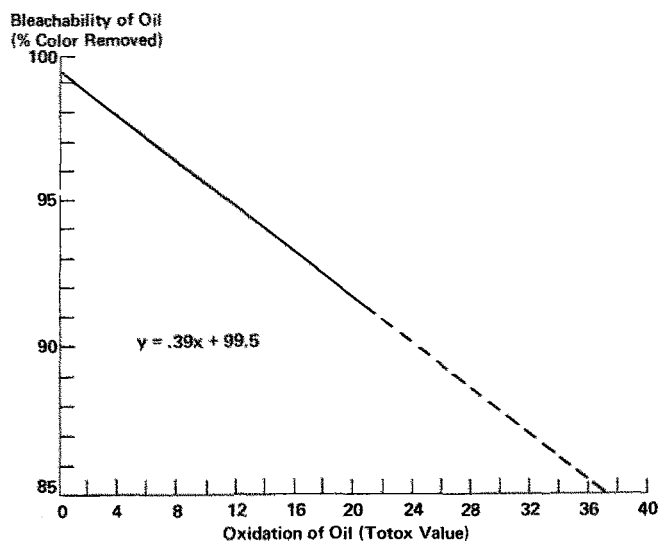


FIG. 7. Effect of oxidation on bleachability of palm oil.

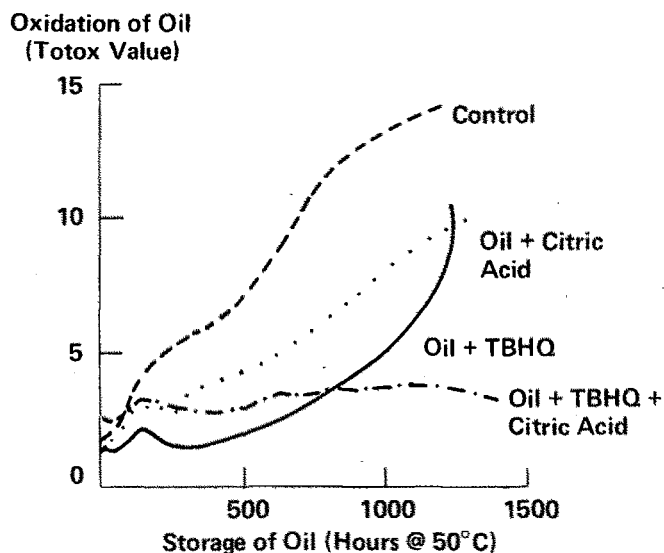


FIG. 8. Inhibition in stored palm oil with TBHQ antioxidant and citric acid treatments.

lead to serious difficulty in refining the oil to good quality. Of major concern is the adverse effect such oxidation has on ability to bleach the oil to the low color levels required to marketing refined, bleached palm oil. As illustrated in Figure 7 (14), oxidation of palm oil, as measured by build-up of TOTOX value (calculated as 2 x peroxide value + anisidine value) such as may occur during shipment and storage of the crude oil, leads to substantial reduction in bleachability, thus making it difficult and costly (in terms of added processing expense) to meet color specifications established for edible palm oil in the world marketplace.

Figure 8 shows data reported by Pike (15) resulting from his evaluations of antioxidants and metal chelating agents in crude palm oil. This demonstrates how antioxidant-chelating agent treatments, such as the TBHQ-citric acid combination shown here, may be effective in suppressing crude palm oil oxidation during storage and shipment.

Subsequent laboratory and plant-scale tests have now been completed (16) to quantify optimum levels of TBHQ and citric acid necessary to achieve the best overall protective effect in crude palm oil. This has been identified as combined treatment with 110 ppm TBHQ plus 365 ppm

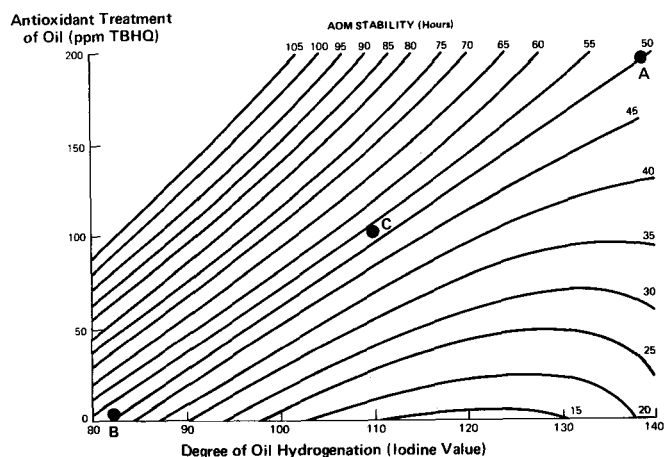


FIG. 9. Effects of antioxidant treatment and hydrogenation on oxidative stability (AOM) of soybean oil.

citric acid which, added to the crude oil as soon as possible after extraction, may be used quite beneficially in palm oil production to: (a.) enable meeting bleached color specifications for refined palm oil; and (b.) save on bleaching costs because of reduced requirements for extra clay needed in bleaching of oxidized crude oils.

Refining of crude fats and oils is done to remove gross nonglyceride impurities consisting mainly of free fatty acids and mucilaginous substances, but with major objectives (17) of accomplishing this with least possible damage to the neutral oil and tocopherols (in vegetable oils) and with minimum refining loss. It is especially important that conditions conducive to oxidation be eliminated (18) because of the readily demonstrable harmful effects that oxidation during refining will have on quality of the finished, deodorized oil. For example, it's estimated (19) that an increase in peroxide content of only 1.2 meq./kg. in neutralized soybean oil may reduce by about 50% the time required for oxidation to occur in the oil after it is deodorized. Oxidation during refining can be minimized mainly by avoiding contact of the oil with air (i.e., through use of hermetic separators) and by eliminating to the maximum extent prooxidant metallic contaminants (especially, copper and iron) naturally present in the raw materials or which may be introduced from processing equipment. With good vegetable oil refining techniques, there may not appear to be justification for adding antioxidants to crude oil during the refining process, especially since vegetable oils at this stage are rich in natural antioxidant (tocopherol) content. However, animal fats do not have the benefit of such natural antioxidant protection, and this is likely the basis for observations of beneficial effects from adding antioxidants to animal fats during refining. Finally, it should be noted there is evidence that antioxidant compounds, including tocopherols, are extracted and removed to some degree from fats and oils during the refining operation.

Bleaching (often considered just a part of the refining process) is the physical, or sometimes chemical, treatment of fat or oil to remove or reduce color (7). Major factors influencing bleachability of oils (20) are type and amount of bleaching clay used, method of bleaching, and quality of the oil to be bleached. Under this last factor, likely having more overall importance than the other two, the oxidative state of the oil has great bearing on ability to remove undesirable color from it, as has been recognized and reported by various investigators (20, 21, 22). Most recently, as described earlier in this presentation, the adverse effects of oxidation on bleachability of palm oil have been reported (14, 23, 24), while the potential quality and

economic benefits from protection of crude palm oil with a potent antioxidant, such as TBHQ, have been demonstrated. Finally, the importance of avoiding contact of oil with air during and immediately following bleaching cannot be overemphasized. Bleaching is commonly done under vacuum to accomplish this.

Hydrogenation is done primarily to harden fats and oils by saturating the double-bonds, but increased oxidative stability and color improvement are definite additional benefits from this process (7). Also, with those oils containing substantial amounts of linolenic acid, or possibly other fatty acids with more than two double-bonds in their molecular structures, there is a unique form of oxidative degradation known as "flavor reversion." With such oils a very selective, light degree of hydrogenation is applied (25, 26, 27) to reduce the trienes and thus minimize the reversion. Phenolic antioxidants are found to be ineffective in inhibiting this type of oil oxidation; however, the use of metal chelating agents and protection of the oil from light (especially ultraviolet) are effective since reversion is strongly catalyzed by prooxidant metals and light.

Of major concern in hydrogenation is catalyst poisoning, and there are indications that such poisoning may result from adsorption on the catalyst of oil oxidation products. It has been reported (28) that when the peroxide value of oil subjected to deodorization exceeds 30 meq./kg., there becomes a noticeable loss of activity of hydrogenation catalyst; however, it may be quite unusual for oil with such advanced oxidative deterioration to be subjected to hydrogenation.

As previously mentioned, increase in oxidative stability is often an important objective of hydrogenation. However, in past years there has seemed to be relatively little interest in comparisons of oxidative stability provided by hydrogenation as compared with that obtainable through treatments with phenolic antioxidants. Also, the possible merit of using hydrogenation and antioxidant treatment jointly to obtain optimum stability benefits with possible economic savings was apparently unexplored. Interest in such antioxidant treatment as an alternative or supplement to hydrogenating oils for increased oxidative stability peaked in the past few years for a number of reasons: (a.) a new antioxidant (TBHQ) with high effectiveness in those oils likely to be hydrogenated had become available for use in food oils; (b.) concern was mounting over availability of hydrogenation capacity, the "energy crisis" was upon us, and hydrogenation costs were increasing rapidly.

These reasons provided the incentive, and, through extensive specially designed experimental programs, we have been able to develop relationships (Figure 9) between effects of antioxidant treatments and hydrogenation, individually and in combinations, on oxidative stability [by AOM (5)] of vegetable oil. As indicated by Point A, the maximum (200 ppm) TBHQ treatment of the unhydrogenated oil resulted in stability of 50 AOM hr. To achieve this same degree of oxidative stability by hydrogenation alone, the iodine value of this oil had to be reduced all the way down to about 80, as indicated by Point B. By means of this response surface (or Contour graph), estimates can be made readily on stabilities to be achieved with combinations of the two treatments. For instance, in this oil, treatment with 100 ppm TBHQ antioxidant of the oil hydrogenated to 110 iodine value provided ca. 50 hr. AOM stability, as indicated at Point C.

Of considerable potential value in fat and oil processing are the cost-effectiveness relationships that may be established based on this type of stability response surface. First, relationships between costs of hydrogenation and antioxidant treatment must be established as illustrated in Figure 10. The derivation of this graph is described in detail in published literature (29), but in general the diagonal lines

represent equivalent costs with respect to variable combinations of hydrogenation and antioxidant treatment. Here, for example, Points A and B represent equal cost for antioxidant treatment (slightly under 100 ppm in this case) and hydrogenation (reduction of iodine value to about 125). Costs of combination treatments can be estimated from this graph as exemplified by Point C indicating, in this instance, a cost of about \$2,800/million pounds of oil for 100 ppm antioxidant and hydrogenation to 110 iodine value.

To estimate degrees of oxidative stability obtainable at various costs of antioxidant treatment and/or hydrogenation, the cost comparison graph is simply superimposed over the stability response surface as in Figure 11. These stability results and cost estimates, compared in this manner, demonstrate the potential value of using an antioxidant such as TBHQ as an alternative to hydrogenation where oxidative stability improvement in oil is the prime objective. This would appear to be a valuable tool in fat and oil processing.

Deodorization of edible fats and oils is primarily a steam distillation method designed for efficient removal of substances present in refined oils in concentrations of perhaps less than 0.5% and which contribute undesirable odor and flavor characteristics. These substances must be removed to provide the stable, bland fat and oil end products which are so much in demand. From the standpoint of oxidation in the deodorization process, probably the most critical factor is that at the high deodorizing temperatures (usually over 200 C), it is critical that air be prevented from contacting the fat or oil since oxidation would be very rapid under these conditions (7). To prevent this from happening, deodorization systems commonly are designed (30) so that the oil is deaerated to remove entrained air prior to being heated to deodorization temperatures. The deodorizer itself should be designed to prevent any leakage of air into the deodorizing oil, and usually there is provision for cooling the deodorized oil before allowing it to contact air. Also, (31) in view of the strongly prooxidant effects of certain metal on oil oxidation, deodorizer construction usually is such that only stainless steel is used to fabricate those parts that will contact the oil at high deodorization temperatures. In some continuous deodorization systems, the oil is kept under inert (nitrogen) atmosphere up to its final packaging as a means of eliminating oxidation tendencies.

It is important to recognize (32) that if oils have oxidized significantly before deodorization, they probably will have poor stability after deodorization. This likely is due to the fact that some (probably odorless and tasteless) oxidation products formed prior to deodorization are not removed, and they carry through into the deodorized oil where they serve to initiate and propagate further oxidation. Also, natural antioxidants (mainly tocopherols) may have been destroyed by oxidation, hence are not present in the deodorized oil. It is observed that where oxidized oils have been deodorized, addition of antioxidant to the deodorized oil may have little or no stabilizing effect. This conforms with the theory that once the autoxidative process has gained a foothold in fat or oil, it cannot be reversed or terminated by added antioxidants.

It is important that some deodorizers are equipped to meter metal deactivator (usually citric acid solution) into the feedstock and, also, to meter antioxidant solution into the deodorized oil as it is discharged and being sent to storage.

Not only are tocopherols widely distributed and function as antioxidants in the vegetable matter from which edible oils of commerce are extracted, but it is important to recognize that high proportions of these "natural" antioxidants survive the oil processing stages, including deodorization, and end up in the finished oils (33) at levels as high

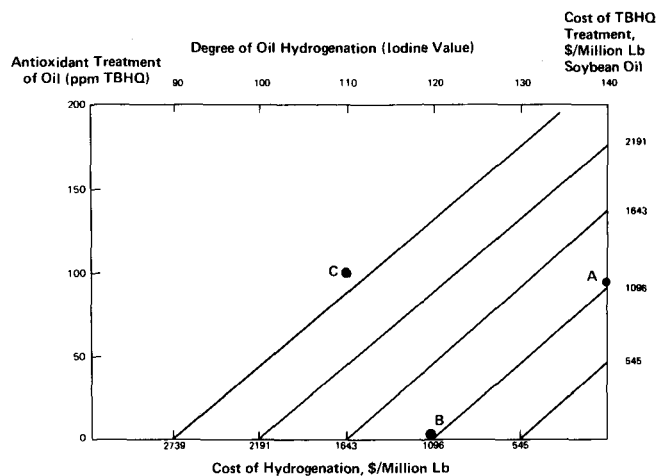


FIG. 10. Hydrogenation and antioxidant treatment costs for million pounds of oil.

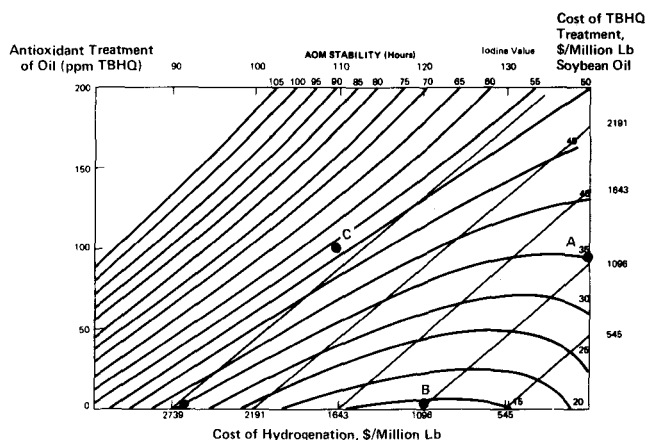


FIG. 11. Oxidative stability (AOM) of soybean oil obtained at various costs for antioxidant treatment and hydrogenation.

as 500-1000 ppm. These residual tocopherols are largely responsible for the oxidative stability inherent in finished vegetable oils. However, it should be recognized that these tocopherol levels seem to be optimum for providing oxidative stability, and addition of more likely will provide no further improvement in stability. If added at high enough concentration, they may even have a depressing effect on the oxidative stability of the oil. However, further improvement in stability of finished oil may be achieved through addition of one or another of the approved "synthetic" antioxidants.

Finally, in reviewing oxidation and antioxidant aspects of the various fats and oil production processes, mention should be made of *fractionation*, which is carried out to provide useful new products and sometimes to rid fats and oils of unwanted minor components. Recent studies reported (34) on palm oil crystallization indicate that oxidation products in palm oil exert significant adverse effects on its crystallization properties, leading to the conclusion that for an oil that is to be fractionated, both hydrolysis (since free fatty acids also adversely affect fractionation) and oxidation must be kept to a minimum.

CONSIDERATIONS IN ACHIEVING BEST RESULTS WITH ANTIOXIDANTS

While various benefits to be realized from the presence of antioxidants in fats and oils during processing become readily apparent, commercial antioxidant applications are

mainly to the finished, fully processed fats and oils with the major objectives of giving them extended shelf life or with the intent that there will be a "carry through" effect resulting in improved stability characteristics imparted to foodstuffs prepared with the stabilized fats and oils. The potencies of the available antioxidant compounds in accomplishing this are extensively documented in the literature (1, 2, 3, 4, 33, 35, 36); however, achievement of the full benefits they have to offer is highly dependent on their proper addition to fats and oils. We continue to observe that the vast majority of difficulties encountered with the use of antioxidants can be traced to improper application techniques resulting mainly from lack of understanding of basic nature of fat oxidation and of certain characteristics of the antioxidants. Possibly the best way to conclude this discussion of oxidation and antioxidants in fats and oils is with a brief review of the major types of problems encountered with the use of antioxidants in edible fats and oils.

Incomplete dispersion of antioxidant in fat or oil results from inefficient mixing equipment, or poorly conceived mixing procedures, or from shortcuts being taken in established procedures for proper antioxidant application. For example, it is unlikely that concentrated antioxidant solution (formulation) simply poured into a storage tank or shipping vessel as it is being filled will disperse sufficiently throughout the fat or oil. This can result in serious stability deficiencies and other complications relative to insufficient antioxidant in the bulk of the oil and unduly high concentration of antioxidant in the oil at the bottom of the vessel. The absolute necessity for complete antioxidant dispersion in the fat or oil must be recognized and necessary steps taken to insure it.

Improper antioxidant concentration. Maximum concentrations of antioxidants permitted in food fats and oils are defined by appropriate government regulations. The approved levels generally are optimum for providing good stability results. A fat or oil processor should realize that the degree of effectiveness of antioxidant within the permitted concentration range is in fairly direct proportion to the concentration used. Accordingly, it may be false economy to reduce treatment levels substantially below the permitted levels with the distinct risk of having fat or oil with inadequate shelf life.

Incompatibility with fats and oils of certain antioxidant formulations. Because of dissimilar solubility characteristics of primary antioxidants and synergists, solvents (propylene glycol, for example) essential in the preparation of good antioxidant-synergist formulations are in some instances not very compatible with fats and oils. In the use of these particular types of formulations, this factor must be taken into account, and appropriate application methods must be used to overcome dispersion difficulties that such formulations may present.

Changes in antioxidant formulations. The many proprietary antioxidant-synergist formulations available for fat and oil stabilization vary in complexity of composition depending upon their active ingredient contents and solvent systems needed to dissolve the ingredients and keep them in solution until the formulation is used. Users of these formulations must keep alert to the possibility that under some storage or handling conditions, separation or crystallization or other changes may occur. This could necessitate special handling to insure proper treatment of fat or oil with any formulation in which such changes are observed.

Use of incorrect type of antioxidant. There are substantial differences between antioxidant compounds and formulations with respect to their performance characteristics in different types of fats and oils and under different processing and handling conditions. Users of fat and oil

antioxidants must give due consideration to these differences in attempting to select the type of antioxidant or formulation which will best fill the particular needs.

Antioxidants added at the wrong stage. Primary antioxidants are very effectively removed from fats and oils during some steps in their production, such as alkali refining, bleaching, and deodorization. While there may be special reasons to add antioxidant to fat and oils at some early state (to crude oil, for example), processors should recognize that a significant portion of the added antioxidant may be removed and not end up in the finished product which may then have insufficient stability because of low antioxidant content.

Failure to add antioxidants soon enough. Since fat and oil antioxidants function by inhibiting free radical formation, the initial step in glyceride oxidation, it is essential that they be added as soon as reasonably possible during production or in the finished product. Rancid oils cannot be rejuvenated by the addition of antioxidants.

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