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Antioxidants and Synergists for Edible Fats and Oils

By
A. R. PRATER.

The subject of antioxidants in fats and oils is so broad that a hurried survey of some of the more general aspects is all that can be accomplished in a brief space. The practical aims of work on antioxidants are, of course, to ascertain whether addition of minute amounts of suitable substances to edible materials, which undergo undesirable changes in flavour owing to attack by atmospheric oxygen, will restrain such attack; what substances have this capacity for retarding oxidation in a marked degree; and especially to discover substances which are non-toxic, soluble or dispersible in the medium to be protected, and free from any deleterious effect on flavour, odour and colour.

Oxidative Rancidity.

Despite many years of research work, the chemistry of oxidative rancidity of fats and oils is still incompletely understood. Notwithstanding the enormous amount of attention devoted to the subject, it still causes losses amounting to millions of pounds annually.

Measurement of Oxidative Rancidity.

Space permits only a brief survey of the more general methods in use for measurement of rancidity. Different methods have yielded varying results and their relative merits are still subject to controversy.

The most practical assessment of oxidative rancidity is by tasting tests by a panel of trained observers. While development of off-flavours is the ultimate criterion of the onset of rancidity, organoleptic tests necessarily lack the precision of chemical tests. Relatively rapid deterioration sets in at the close of the "induction period" and the detection of this point serves as a measure of the stability of the fat or oil. The "induction period" is a period when the absorption of oxygen is relatively small. The line of separation between the period of slow absorption and more rapid absorption is not necessarily a clear one for all fats, e.g. some vegetable oils. The chemical methods include estimation of peroxide oxygen, aldehydes and direct measurement of absorbed oxygen, each of these three determinations being carried out in a variety of ways. The relationship between results obtained by these methods and by organoleptic tests is variable, and it is necessary to specify the test conditions in detail before useful conclusions can be drawn. To save time, accelerated tests have been developed, but every precaution must be taken in the interpretation of these results for they need not necessarily bear any relationship to natural deterioration.

Mechanism of Autoxidation of Fats and Oils.

As was stated earlier, no all-inclusive theory of autoxidation can yet be formulated. Once the induction period is past, the chemical reactions occurring are rather variable and are, in part, dictated by the nature of the reaction products themselves. Many factors may facilitate the acquisition of a critical increment of energy by fat molecules in the average state. The agencies may be mechanical, such as collision of molecules with the walls of the vessel; physical, such as light or heat; and chemical, such as metals like iron and copper. The energy acquired not only promotes the reaction but results in the formation of a highly reactive molecule whose excess energy is passed on to another average molecule. The autoxidation has some of the typical characteristics of an autocatalytic reaction.

Classification of Antioxidants and Synergists.

In an early paper by Olcott and Mattill (1936) nitrogen-free inhibitors of fat oxidation were classified into three groups:

1. Acid type.
2. Inhibitols and hydroquinone.
3. Other phenolic inhibitors.

This classification has since been tentatively simplified by Mattill (1945) as follows:

1. With few exceptions the only substances which have primary antioxygenic action on fatty acids are ortho and para di- and polyphenolic compounds or substances having similar electronic configuration.

2. All other substances, which delay or inhibit the autoxidation of fats, should properly be called synergists because they merely reinforce the effect of the phenolic compounds present and have little, if any, activity apart from them. These are usually di- and poly-basic acids.

Natural Antioxidants and Synergists.

It was not until 1931 that any attempt was made to correlate the induction period with the presence of natural antioxidants. The first reference to this was made by Mattill (1931).

It is necessary here to distinguish between the relative stability of naturally-occurring fats and the relative instability of highly refined fats, which are present in edible products of manufacture. The two main natural fat stores, animal tissues and vegetable seeds offer an instructive analogy to the two methods of protection from oxidation, namely, the exclusion of oxygen or the addition of a protective substance. Animal body fats are for the most part relatively deficient in natural antioxidants, yet they are perfectly stable and remain free from oxidative rancidity *in situ*, because there is practically no access of molecular oxygen to the adipose tissue. On the other hand, vegetable seed fats may be exposed to air but are relatively stable due to the presence of naturally occurring protective substances. From this a fundamental principle in stabilization can be drawn, namely, that if the fat in question has naturally an optimum content of some phenolic antioxidant, the addition of further amounts may be useless; it may even be detrimental, as in the case of tocopherols, because they also are subject to oxidation; the quality of the fat may be depreciated by the oxidation products of phenolic inhibitors. Such fats can be benefited by the addition of synergists which prolong the action of the phenolic stabilizer. In the same way the addition of a phenolic inhibitor to an animal fat should not be overdone. It is wise to add as little as necessary and to reinforce what is added by the simultaneous addition of synergists alone or in combination.

Kinetics of Antioxygenesis.

The following suggestions have been put forward by Calvin Golumbic (1946): There is a latent period called the induction period, during which the absorption of oxygen and the accumulation of peroxides are slow, followed, sometimes rather abruptly, by a period of increasingly rapid oxidation. The induction period is generally attributed to a chain-breaking reaction between the activated peroxides (the corners of the chain) and the inhibitors present. Disappearance of the antioxidant results in the end of the induction period. In the proposed synergistic mechanism the assumption is made of an oxidation-reduction reaction between the synergist and the oxidized form of the antioxidant, whereby the latter is maintained in a reduced form as long as there is synergist available to furnish the hydrogen.

In a paper such as this, it is not possible to mention the vast number of substances which have been used as antioxidants or synergists, nor is is possible to mention here the variety of edible fats and oils which have been treated with these protective substances.

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Projected Survey of Food Canneries in Australia

Plans are at present being made by C.S.I.R., Division of Food Preservation, to visit selected canneries in Australia and carry out a survey with the following objects:

1. To secure first-hand information on the problems with which the canning industry is confronted.
2. To define as precisely as possible the actual starting and finishing dates for each product processed by canneries engaged in the seasonal production of various canned foods.

The main purpose of the survey is to endeavour to assist the canning industry by the subsequent investigation in the laboratories of this Division of problems common to processors and also to determine trends with respect to the future development of the industry.

In the meantime suggestions regarding the nature of the survey would be welcomed, and these should be forwarded to The Chief, C.S.I.R. Division of Food Preservation, Private Bag, P.O., Homebush, N.S.W.

"Pink Whites" in Stored Eggs

By
W. J. Scott.

The term " pink whites " has been applied to a particular defect in shell eggs which have been stored for some time. The condition is not apparent before storage, but its development during storage is not associated with microorganisms and from a bacteriological aspect the egg contents remain wholesome. The abnormal and unattractive appearance, however, renders the eggs unmarketable and losses of up to 10 per cent. have been attributed to this type of disorder. During many storage experiments carried out over the last eight years in Australia the defect has been noted only in occasional eggs, but in the last season's export pack reports from London indicated that certain consignments contained up to 3 or 4 per cent. of eggs affected in a similar way. The purpose of this note is to summarize the information at present available regarding the causes and development of the " pink white " condition.

There are several features characteristic of the condition which may be manifest in varying degrees of severity. The whites may be coloured faint pink to reddish, or at times have a yellowish or amber tint. At ordinary temperatures the yolks are rather watery, enlarged, and frequently of a salmon pink colour. Yolk colour may, however, be normal. At low temperatures the yolks have a marked clay-like consistency, and when cooked they become rubbery. The eggs are free from rot-producing bacteria and have no abnormal odour. Other features of eggs affected in this way are that the yolks have an increased water content, a lower than average fat content on a dry weight basis, and the pH values of the white and yolks tend to be close together at about 8·4 to 8·5. All the above changes are consistent with an increased permeability of the yolk membrane and Schaible and co-workers have produced further evidence on this point in a series of papers published recently (Poultry Science 25: 440-59, 1946). These workers have shown that, in the abnormal eggs, albumen from whites had actually diffused into the yolk, and several yolk constituents had diffused into the white. The pink colour of the whites was shown to be due to traces of iron which had diffused from the yolk and, according to these authors, combined with one of the proteins in the white, the conalbumin.

The cause of " pink white " development is now well established and is undoubtedly due entirely to the diet of the hens. The significant material is found especially in the oil portion of the seeds of plants belonging to the family Malvaceæ. This family includes such economic plants as cotton, weeds such as " mallow " or " marshmallow " (*Malva parviflora*) and *Sida retusa*, and garden plants such as Hollyhock and Hibiscus. More than 20 years ago several investigators in the southern portion of the U.S.A. reported that rations containing cotton-seed meal resulted in eggs with a poor storage quality. Some of these rations contained more than 30 per cent. of cotton-seed meal and, in such cases, deterioration was rapid and widespread. These studies showed that up to 4 or 5 per cent. of cotton-seed meal could be incorporated in the ration without any measurable loss of storage quality, whereas additions of 10 per cent. meal resulted in considerable deterioration.

In 1933 knowledge of the subject was advanced considerably by Almquist and Lorenz (U.S. Egg and Poultry Mag., April, p. 28), who, in addition to making a detailed description of the defect, showed that the fat content of the affected yolks gave a positive Halphen test. This test is a colour reaction obtained with a minor constituent of unrefined cotton-seed oil and is used for the detection of this oil. As shown by these authors, the test is given also by oil extracted from seeds of other Malvaceous plants. The pink white condition in stored eggs was induced readily by feeding hens with rations containing 2 per cent. of crude cotton-seed oil. Likewise feeding seeds of the weed *Malva parviflora* caused the yolk fat to become Halphen-positive and after a few weeks the development of "pink whites" in the stored eggs.

More recent work by Lorenz (Poultry Science 18: 295, 1939) and by Schaible and others (*ibid.*) indicates that cotton-seed meal contains two factors causing deterioration. The first is gossypol, which causes olive yolk formation, and this disorder can be prevented by the addition of soluble iron salts to the ration. The second factor is a compound closely associated, or identical with, the Halphen substance, and this gives rise to the "pink white" condition. The second factor is believed to be the only one common to other Malvaceous seeds. It appears also from Schaible's experiments that the influence of cotton-seed rations is merely one of accelerating a normal change. Eggs produced by the hens fed on cotton-seed meal or oil usually developed "pink whites" within 3 or 4 months' storage, but eggs produced by hens which had no access to cotton-seed materials slowly developed the "pink white" condition after storage for 2 to 3½ years. For practical periods of storage not exceeding say 8 to 9 months, it is clear, however, that "pink whites" will only develop when the hens have had access to certain materials produced by malvaceous plants. The elimination of these materials from the diet is at present the only known method of combating the trouble. At the present time some preliminary experiments are being undertaken in these laboratories to determine whether other methods of control may be feasible.

While the "pink or amber white" defect noted in the examination, in England, of a number of consignments of Australian eggs has not yet been proved to be due to the ingestion of malvaceous seeds or seed-products, nevertheless the similarity of the symptoms of this defect with those described by the American investigators points to a possible common causation. Pending the results of investigations in Australia, it would, therefore, be a wise precaution for poultry farmers to deny hens an access to stands of mallow plants and to avoid the use of cotton-seed meal in mashes fed to the birds.

Apricot Juice and Apricot Nectar

By
P. THOMPSON.

During recent years the canning of pulpy juices, prepared from stone fruits, has grown rapidly, one of the fruits meeting with remarkable consumer acceptance being apricot.

Full flavour for this type of product is not developed until the apricots reach the soft ripe stage. They are usually packed as straight juice or as a diluted and sweetened product, variously described as " beverage ", " cocktail " or " nectar ".

The fruit should be tree-ripened, and, for a first-quality product, three or four pickings should be made to get the best flavoured fruit.

The American procedure for making apricot juice is to steam washed and pitted apricots until soft, to pass them through an expeller screw extractor of the type commonly used for making tomato juice, to add one part of sugar to three of pulp, to can in plain tin cans, to exhaust for 8 to 10 minutes (301 × 411 cans), to process for 20 to 25 minutes at 212° F., and finally to cool immediately. The undiluted juice prepared in this way is of purée consistency, and requires dilution with water before use as a beverage. (Tressler, Joslyn and Marsh, Fruit and Vegetable Juices, 1939, p. 285.)

The other beverage is a ready-to-drink product which is prepared by diluting apricot juice with syrup before canning. Shallah and Cruess suggested the following procedure which is widely used in American commercial production (*ibid.*, p. 286).

" Whole apricots are washed thoroughly in cold water. They are pitted and steamed until cooked through. This heating kills the oxidase and facilitates the pulping. Ordinarily the heating need not exceed five minutes. The hot fruit is run through a brush finisher or tomato pulper equipped with screens of about one-millimetre holes. It is possible to do the pulping and separate the pits and skins by a single operation, although this depends on the efficiency of the machine and on the degree of ripeness of the fruit.

" The purée is sweetened with about an equal volume of cane-sugar syrup of about 15° Balling. The resulting diluted, sweetened purée is filled into plain tin cans, exhausted for six minutes at 212° F., sealed and cooked for 15 minutes at 212° F. Large sizes should receive a longer processing period since the heat penetration through purée is rather slow."

This laboratory has prepared apricot " nectar " from local fruit with very good results. The nectar was acidified slightly, which gave the beverage a slight " bite " which considerably enhanced its palatability.

The procedure followed was carefully to sort the fruit and eliminate the green and moulded fruits, remove the pits and pass through a disintegrator (a type of vertical hammer mill equipped with a fine screen with $\frac{1}{16}$ inch diameter holes). To the purée from this operation was added an equal volume of syrup, 12 per cent. Brix, containing 1 per cent. citric acid, which gave a final concentration of 0·5 per cent. added acid. The nectar was then heated to 160° F., filled into plain cans (301 × 411), sealed, processed for 20 minutes at 212° F., and finally cooled.

Rust Prevention on Cans

Results of tests on a procedure for cleaning and desensitizing cans for the prevention of subsequent rusting are described by R. S. Gillies in "Food, Processing, Packing, Marketing" 16: 81, 1947. The tests carried out were based on the process developed in the research laboratories of the International Tin Research and Development Council, involving the use of sodium dichromate in alkaline solution for desensitizing cans which had been internally treated with a view to protection against sulphide staining. The experiments reported by R. S. Gillies were made to ascertain the commercial application of this process with particular reference to the treatment of the outside of the can and especially to determine whether such treatment could be applied during the actual processing time, thereby eliminating the usual commercial practices of passing processed cans through a washing machine containing the detergent and then through the dichromate solution before lacquering.

Vertical pressure-cooling retorts were employed since it was necessary totally to immerse the cans in water during processing. In order to reduce the adherence of precipitated calcium and magnesium salts to the tinplate it was found necessary to reduce the hardness of the water by passing it through a base exchange water-softening plant. Even after such treatment of the well water used a soft deposit adhered to the cans after processing, but it was easily removed by light rubbing. This deposit was most noticeable when sodium carbonate, sodium metasilicate and trisodium phosphate were the detergents employed. Sodium hexametaphosphate gave the best all-round results, the adhering film being so thin as to be of little consequence. Several common detergents in varying strengths were tested under the working condition of the retort in which cans were immersed in water containing the detergents, with or without dichromate in varying concentrations. The processing time and temperature selected was 240° F. for $2\frac{1}{2}$ hours.

The procedure was as follows: Sufficient water was added to the retort to immerse the cans, and the alkaline chromate stirred in. At the end of the processing time chlorinated softened cooling water was allowed to enter at the bottom of the retort until the overflow was cold. The bottom exhaust valve was then opened and the water run off. The final cooling was obtained by means of spray jets in the lid of the retort, which helped to rinse off the fine white deposit on the cans. If removed from the retort while still sufficiently warm, the cans were dry and ready for lacquering.

Tins treated by the procedure outlined were exposed for three weeks on the factory roof and examined for evidence of rusting with the following results:

Untreated tinplate showed signs of rusting within a week.

Tinplate treated with concentration of sodium dichromate lower than 0·1 per cent. showed some sign of rusting after about two weeks.

Tinplate treated with concentrations of sodium dichromate of 0·1 to 1·0 per cent. showed little signs of rusting after three weeks' exposure. A summary given by the author is as follows:

1. The successful application of a film of lacquer on cans depends upon the cleanliness of the tin surface. The degreasing may be accomplished during processing by total immersion of the cans in a detergent solution of suitable strength, using a vertical pressure cooling type of retort.

2. Electrolytic action will bring about feathering or will entirely strip the tin (depending upon the detergent employed) owing to the cell set up between the tinplate and skips, which are of mild steel construction.

3. The addition of a small quantity of sodium dichromate will prevent feathering.

4. Sodium dichromate serves a double purpose, since it desensitizes the metal surface and therefore will prevent rust formation.

5. Cans which have been processed in an alkaline sodium dichromate solution may, as soon as dry, be sent to the lacquering plant or labelling machines without further cleaning.

6. The strength of the detergent required will depend largely on the initial cleanliness of the can as received from the filling and closing machines. If due care has been taken and a minimum of fat and food particles is left adhering to the tin surface, then a 0·1 per cent. solution of the common detergent, together with 0·1 per cent. of sodium dichromate, will give satisfactory results. It will be found beneficial if the cans receive a preliminary washing by means of a can washing machine immediately after closing in order to remove the more resistant particles of foodstuffs by mechanical action.

Further details of the experiments carried out by the author are given in the article published in " Food, Processing, Packing and Marketing ". No estimate is given of the probable cost of processing cans by this method.

Frozen Strawberries

Both frozen and dehydrated strawberries are liable to develop a peculiar hay-like odour during storage, said to be due to the formation of coumarin. With the dehydrated product it has been found that packing in cans in an atmosphere of nitrogen prevents the formation of the odour, which is presumably the result of oxidative changes. An analogous process—vacuum packing in cans—is now being applied to frozen strawberries in the U.S.A., and is said to be effective in eliminating the hay-like odour. Sliced strawberries are packed in 60 per cent. sugar solution in No. 10 cans, sealed in a vacuum sealing machine (10-inch vacuum) and the cans frozen.

Canning on U.S.S.R. Fishing Trawler

New all-welded trawlers of 1,500 tons displacement are being designed by Leningrad engineers for the Soviet fishing industry. The vessels will not only be exceptionally seaworthy but will have the most up-to-date equipment, including hydro location and sounding installations for locating fish shoals. The holds will contain equipment for canning fish and the production of fish flour and cod-liver oil.

In the course of a 20-day trip, such a trawler can manufacture 10,000 tins of cod's liver, 30 tons of fish flour, 100 tons of salted and frozen fish, and 10 tons of cod-liver oil.

Under the Five-Year Plan the Soviet fishing industry will be equipped with 150 trawlers of this type.

Processing Food for Sterilization

In a series of three articles which appeared in "Food Industries" 19: Nos. 1, 2 and 3, 1947, C. Olin Ball discusses fully the principles of heat sterilization in canning and deals particularly with the complex behaviour of steam and air mixtures. He also classifies and discusses five different types of high-short sterilization methods aimed at improving canned foods whose quality is impaired by the ordinary sterilizing processes. In dealing with the requirements of a satisfactory process its merits should be assessed from consideration of four factors: (1) Economy; (2) Quality of product; (3) Uniformity of product; (4) Sterilization of product. In his discussion of heating food in containers the following methods are listed by the author:

1. Heat applied to only one face of container.
2. Heat applied to multiple faces of container.
   - (*a*) Container enveloped in heated air.
   - (*b*) Container enveloped in mixture of air and steam.
   - (*c*) Container enveloped in pure saturated steam.
   - (*d*) Container enveloped in heated water.
3. Container subjected to influence of high-frequency electrical waves.

Method 1 has never received serious consideration in connection with canning techniques.

Method 2a as exemplified by a container within a heated oven has been used in home canning but it can be disregarded for commercial canning since it is slow and potentially dangerous.

Method 2b has been used commercially in processing glass containers in retorts, but it has lost favour because of the difficulties in control in retorts of commercial size. Despite the rejection of this method, the functioning of a mixture of steam and air as a heating medium still merits thorough study since even in pure steam processing in still retorts the effect of steam-air mixtures must be taken into account. Regions commonly called "air pockets" containing mixtures of steam and air are always present in a retort for some time after steam is first turned into the retort, and they persist until all air has been expelled from the retort by venting. Entrapping of air in certain regions within the retort is partly due to the fact that steam inlets and vents are not properly located to produce movement of steam into all parts of the retort and is also due to retardation of steam circulation by containers, trays, etc. In practice the dangers of localized formation of "air pockets" and the resulting lowering of temperature in the affected regions are reduced by provision for venting the retort throughout the whole period of heat processing. After a theoretical discussion of the attainment of equilibrium conditions the author emphasizes that the only possibility of maintaining a uniform temperature throughout the retort in a steam-and-air-process must be in producing a violent circulation of the vapours in the retort at all times ; in other words the circulation must be sufficiently rapid to overcome all variations. A very small quantity of air in the steam supplied to the retort may be sufficient to build up around a container of food an air blanket of sufficient density materially to reduce the saturation temperature of the steam in contact with the container. The author puts forward this hypothesis to explain the phenomenon of "broken" heating curves in which there is an abrupt change in the rate of increase of temperature within a container of food where there is no apparent change in the procedure of applying heat.

Method 2c is the one universally used for containers that do not require retort pressure greater than the saturation pressure of steam at processing temperatures. A schematic drawing of a commonly used set up for vertical retorts employed for processing in steam is given in No. 3 of the series.

Method 2d is the accepted method for processing containers that require retort pressure greater than the saturation pressure of steam at the processing temperature. These include glass containers in general because the strength of the grip of container-covers on to the glass containers is not sufficient to resist the force of the internal pressure during the process. Pressure above saturated steam pressure at the water temperature is provided by compressed air in the headspace of the retort. In this method of processing, all containers are immersed in water that is kept in agitation to give good distribution of temperature. This gives uniformity of temperature throughout the retort during all stages of the process. A schematic drawing is given of a commonly used set-up for vertical retorts employed for processing containers in water.

Method 3, employing the use of high-frequency electrical waves, has not yet reached the stage of practical application. The author states that :

"Present indications seem to be, however, that at least until further improvements can be made in high-frequency generation technique, this method of sterilization employing either inductive or di-electric heating, will compare unfavourably in cost with methods which employ steam as a heating medium. The use of ultra high-frequency waves may, however, uncover a new field beyond that of either inductive or di-electric heating."

Dealing more specifically with so-called high-short sterilization, the author quotes the definition given by C. O. Ball (1938) in "Food Research" 3: Nos. 1 and 2, 13-55.

"High-short sterilization of foods is sterilization by heat applied for times ranging from a few seconds to a few minutes. High-short sterilization implies heat penetration so rapid that, to obtain it, the food must either be heated in bulk or in containers that are kept in violent agitation. The maximum length of a process in this category must be arbitrarily chosen. We believe we are justified in stating that a process in which the heat treatment is longer than six minutes is not a high-short sterilization process." The author then goes on to classify and discuss high-short sterilization methods of five types :

(1) Food heated but not completely sterilized before it is put into the can. Containers would be filled with food at 250° F. or higher, sealed immediately, and held long enough before cooling to permit the sterilization of the interior surfaces of the container to be effected by heat from the food.

(2) Food completely sterilized before it is put into the can. There are two variations of this process. In the first, the cans are heated after sealing to destroy bacteria which may have contaminated the food during the filling and sealing operations; in the second, the containers are pre-sterilized and then filled and sealed under aseptic conditions.

(3) Food heated in the can before sealing with sterilization completed after sealing. Under ideal conditions the container should be sealed as soon as the desired sterilizing temperature is reached in the container and the container, before cooling, should be held long enough to permit the food and the interior surfaces of the container to be sterilized by the heat in the food. When the temperature imparted to the food is higher than water boiling temperature at atmospheric pressure, the operations of heating the food and sealing the container must take place under sufficient external pressure to prevent boiling of the food before sealing.

(4) Food heated in the can and completely sterilized before sealing.

(5) Food heated in the can after sealing. A process of this type requires agitation of the container during the heat treatment in order to obtain rapid changes of temperature in the food.

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