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PROCESS FOR IMPROVING OILS

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This invention pertains to the art of improving oils which contain vitamins and more particularly to the treatment of vitamin-containing oils and concentrates to remove substances which cause undesirable taste and odor.

Fish oils and concentrates prepared therefrom are of value for medicinal purposes because of their high vitamin A and/or D content. Likewise, palm, wheat germ and cottonseed oils are valuable pharmaceuticals because of their vitamin E, K or carrotene content. However, as a general rule, they have been unsatisfactory from the standpoint of taste and odor. This is particularly true in the case of the fish oils. Attempts have been made to improve them with chemical treatment but the results have been unsatisfactory, particularly inasmuch as the vitamin was at least partially destroyed.

This invention has for its object to provide a method for effectively removing undesirable odor and taste from vitaminous oils and concentrates thereof. Another object is to provide a method for removing the odor and taste without chemical agents. A further object is to provide a method for effectively removing odor and/or taste from fish oils and fractions thereof which contain vitamins, without causing substantial loss of the vitamin contained therein. A still further object is to provide a cheap and economical process for removing the substances which cause odor and taste. Other objects will appear from the following description and claims.

These and other objects are accomplished in accordance with our invention which includes subjecting the oil or concentrate to vacuum treatment under such conditions that part of the vitamin content of the oils or concentrate distills, but most of it remains undistilled. It has been found that many of the undesirable components are of relatively low vapor pressure but, nevertheless, can be distilled from the oil or concentrate. Although these substances distill somewhat in the same range as the vitamins, it is possible to separate them as a distillation fraction without simultaneous distillation of more than a few percent of the vitamins contained in the oil or concentrate.

During distillation of fish oils to produce vitamin concentrates decomposition products are often formed and condensed in the distillate in amounts which depend upon the condition of the distilland and the distillation procedure. These materials are in a large measure free fatty acids although other substances, such as coloring matter, etc., are present. These substances, generated during a previous distillation, can likewise be removed by our process of partial or incipient distillation.

The incipient distillation is carried out under a high vacuum, that is, between 1 mm and 10^{-6} mm., preferably below 10^{-2} mm. The distilland oil is caused to flow in a relatively thin film over a heated vaporizing surface and the vapors are condensed upon a condensing surface separated from the film by substantially unobstructed space. The distances between the film and the condensing surface is relatively unimportant but short distances such as ½ to 12 inches and especially 1 to 6 inches, are preferred. This distance can be less than the mean free path with advantage.

It is best to regulate distillation conditions so that the undesirable materials are removed without, at the same time, removing any more of the vitamins than necessary. The temperature, time of treatment, and thickness of the film, or degree of attenuation, are the important factors and are interdependent. When the oil is treated while flowing by gravity over the surface of a vertical column, the film thickness is usually about .2 to 1 mm. However it is advantageous to introduce the oil onto a heated spinning disc over which it passes as a thin film by centrifugal force. In such cases the film will be much thinner and of the order of .01–.1 mm. It will be understood that these thicknesses are preferred, but that equivalent or useful results can be secured by thicker films up to a centimeter. Operation with these thicker films requires much larger times of exposure to the vacuum and therefore is not recommended. The centrifugal treatment can be conveniently carried out in a centrifugal still such as described in my copending application 99,632 filed September 5, 1936. This apparatus comprises a heated rotating surface provided with means for introducing distilland onto the center thereof. A condensing surface is located nearby and means is provided for maintaining a vacuum between the two surfaces.

With gravity flow methods and film thicknesses usual therewith the time of treatment will vary between about 10 seconds and 15 minutes. With centrifugal methods the time of treatment can be as high as with gravity methods and may be as low as .01 second. It will be realized that the time required for treatment is more or less directly proportional to the thickness of the film and therefore that the shorter periods can be used with the thinner films. The temperature should

be between about 80° and 220° C. Temperatures of about 140°–180° are most generally useful.

Since the object is to remove the odorous substances without removing the vitamins it is best to adjust the conditions so that not over about 10% of the vitamin distills. The higher temperatures cause more rapid distillation of vitamins and should be used in conjunction with the shorter time periods. In some cases very poor quality oils of no commercial value because of their high free fatty acid content may require the removal of as much as fifty percent of the vitamin before complete removal of the undesired constituents takes place. Fractions containing about .5 to 1 or 2 percent of the vitamin are usually sufficient to remove substantially all of the odorous and other undesirable substances.

The invention is of particular advantage for treating odoriferous fish oils such as tuna-liver oil. However, it is valuable for the treatment of vitamin oils in general, such as wheat germ, cod-liver, halibut-liver, pollack, salmon, swordfish, greyfish, etc. oils and vitamin concentrate oils produced by high vacuum distillation or saponification. The process is of particular advantage for treating preliminary vitamin fractions obtained by high vacuum distillation of fish oils since they often contain the bulk of the odors, tastes and free fatty acids, etc. of the fish oil, in concentrated form.

Example I

A vitamin A concentrate prepared by molecular distillation of rank tuna-liver oil and having a vitamin A content of 194,000 units per gram and poor odor and taste was subjected to vacuum treatment under incipient distillation conditions. This treatment involved passing the oil in a thin film over a heated vaporizing surface of a high vacuum, short path still at a pressure of about .001 mm. The conditions and results are indicated in the table below:

Fraction No.	Temp.	Per cent stripped off by weight	A-units per gram	Per cent A	Acid value
Original			194,000		33
1	110	8.1	225,000	9.4	106
2	120	7.3	200,000	7.5	102
3	130	8.1			98
4	145	7.3			74
5	150	2.1	135,000	1.4	68
Residue			160,000	55	4.8

This was a particularly bad case in which the acid value, etc. was very high at the start. The taste and odor of the undistilled residue improved as the temperature increased. A large portion of the vitamin A was removed, but it was largely of the alcohol or low boiling form. 70% of the more valuable vitamin D remained in the deodorized residue.

Example II

A greyfish-liver oil fraction distilling at 170°–190° under molecular conditions and containing 400,000 units of vitamin A per gram was introduced onto a molecular still vaporizing column and caused to flow in a thin film over the heated vaporizing surface thereof at the rate of 5–10 gal. per 24 hours, the column being 3" x 30". The pressure was maintained at .001 mm. and the temperature at approximately 150° C. All detectable taste and odor was removed as distillate along with approximately 1.5 per cent of the vitamin A content of the original fraction.

The fraction containing the odors etc. may be discarded although it can be treated to recover its free fatty acid and vitamin content.

What we claim is:

1. The process of removing substances which cause undesirable odor and taste from vitamin-containing oils, whereby the odor and taste materials are obtained as a distillate and the purified vitamin oil is obtained as a distillation residue, which process comprises subjecting the oil to short path distillation at a pressure below .1 mm., distilling off from the oil a fraction which contains between .5 and 2% of the vitamin content of the oil, condensing this fraction and withdrawing it from the still and withdrawing the purified oil from the still as undistilled residue.

2. The process of purifying concentrated vitamin A prepared by high vacuum distillation of a fish oil in order to remove substances which cause odor and taste, which process comprises subjecting the concentrate to short path distillation at a pressure below .1 mm. and separating a distillate fraction containing about .5–2% of the vitamin content of the concentrate whereby the undesirable odor and taste substances are removed as distillate with relatively small amounts of vitamin and the vitamin concentrate containing substantially its entire original vitamin content is withdrawn from the still as undistilled residue.

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