



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁴ : A23D 5/00, C11B 3/00	A1	(11) International Publication Number: WO 88/ 02989 (43) International Publication Date: 5 May 1988 (05.05.88)
(21) International Application Number: PCT/US87/02738 (22) International Filing Date: 20 October 1987 (20.10.87) (31) Priority Application Numbers: 921,184 020,613 (32) Priority Dates: 21 October 1986 (21.10.86) 2 March 1987 (02.03.87) (33) Priority Country: US (71) Applicant: GENERAL MILLS, INC. [US/US]; 9200 Wayzata Boulevard, Minneapolis, MN 55426 (US). (72) Inventors: MARSCHNER, Steven, S. ; 2800 Girard Avenue South, Minneapolis, MN 55408 (US). FINE, Jeffrey, B. ; 2925 Raleigh Avenue South, St. Louis Park, MN 55416 (US).		(74) Agents: FRONEK, David, N. et al.; Dorsey & Whitney, 2200 First Bank Place East, Minneapolis, MN 55402 (US). (81) Designated States: AU, BE (European patent), BR, CH (European patent), DE (European patent), DK, FI, FR (European patent), GB (European patent), IT (European patent), JP, KR, NO, SE (European patent). Published <i>With international search report.</i>
(54) Title: PHYSICAL PROCESS FOR SIMULTANEOUS DEODORIZATION AND CHOLESTEROL REDUC- TION OF FATS AND OILS (57) Abstract Physical processes for fats and oils, especially fish oil, involving simultaneous deodorization and cholesterol level reduction. Either freshly refined or stored oils that have oxidized (reverted fish oil) can be treated. In the process the fish oil is deaerated, mixed with steam, heated, flash vaporized, thin-film stripped with countercurrent steam, cooled (all steps being performed under vacuum), and then stored under oxygen free conditions. The process provides fish oils which are clean tasting and from which has been removed substantially all of the free or non-esterified cholesterol.		

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PHYSICAL PROCESS FOR
SIMULTANEOUS DEODORIZATION AND CHOLESTEROL
REDUCTION OF FATS AND OILS

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CROSS-REFERENCE TO RELATED APPLICATION

This application is a continuation-in-part application of copending application Serial No. 921,184, filed October 21, 1986.

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BACKGROUND OF THE INVENTION

1. Technical Field

The present invention relates to food products. More particularly, the present invention relates to methods for treating fats and oils, especially fish oils, for deodorization and cholesterol level reduction. In its product aspect, the present invention relates to edible oils, especially fish oils, characterized in part by bland flavor and reduced cholesterol level.

2. Background Art

A great deal of attention has been given to the various health benefits apparently associated with the consumption of fatty fish. The health benefits appear to be related to the presence of high levels

of the n-3 family of polyunsaturated fatty acids. Oils containing these fatty acids, such as fish oils, are referred to as "omega-3" oils and desirably contain high levels of n-3 fatty acids, especially
5 eicosapentaenoic acid ("EPA") and docosahexaenoic acid ("DHA").

Notwithstanding the present interest, non-hydrogenated fish oil, generally, to date has not been used widely in food products due to problems
10 in odor, flavor and especially stability, primarily oxidative rancidity. Also, fish oil undesirably contains cholesterol. However, canned salmon has had added salmon oil. Also, certain canned meat products have been marketed in Japan which have had refined fish
15 oil added as a nutritional supplement.

Hydrogenated fish oils are much more stable due to the decrease in the degree of polyunsaturation and are widely used in Europe for margarine. Hydrogenation is also effective in reducing odor and
20 off-flavor development after deodorization. However, hydrogenation by decreasing polyunsaturation, including the n-3 fatty acid content, correspondingly decreases the health value of fish oil.

The present invention relates to a physical
25 method which reduces the cholesterol level in fish oils while simultaneously deodorizing them. Reducing the cholesterol content of a fat is also nutritionally important since reducing cholesterol intake can reduce serum cholesterol, and reducing serum cholesterol
30 has been shown to lower the risk of heart disease. Chemical extractant methods for cholesterol removal or reduction in oils are known. Chemical methods are too expensive, however, for commercial scale use. Also, chemical extraction undesirably results in the
35 production of oils contaminated by residual amounts of chemical extractants. While several chemical methods

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for removing cholesterol are known, the only physical method believed known in the art is molecular distillation.

Molecular distillation for cholesterol reduction, however, requires extreme operating conditions including a pressure in the micron range. While molecular distillation is effective for removing some but not all cholesterol, the process can yield a fish oil with undesirable darkening of oil color and an undesirable strong "chemical" flavor. The dark color and off-flavor are due to the presence of odoriferous materials which remain after completion of molecular distillation. Molecular distillation thus still requires subsequent deodorization. The two processes cannot be combined since the vacuum conditions which define molecular distillation preclude the addition of steam which strips off these odoriferous materials. Additionally, commercial scale molecular distillation equipment is very costly. Most importantly from a cost standpoint, another major disadvantage of molecular distillation is the significant product loss which is associated with the "distillate" fraction. In molecular distillation the distillate fraction can run from 7% to as high as 20% or higher in order to achieve cholesterol reduction comparable to the results of the present invention. Since the distillate fraction is unusable due to its containing the undesirable constituents in concentrated form, such high distillate fractions reduce overall yields. Reduced yields is a most significant cost factor due to the high cost of the starting material in addition to the high cost of molecular distillation itself.

Surprisingly, another physical method has been discovered for removing substantially all of the free or "non-esterified" cholesterol from fish oils whereby the overall cholesterol level is reduced

and which overcomes many of the disadvantages of molecular distillation. In the principal operative step, the operating conditions utilize a much higher pressure than employed in molecular distillation, so as to maintain steam during stripping. Surprisingly, these conditions are nonetheless effective for cholesterol removal. Furthermore, the equipment needed to practice the method is commercially available on a commercial scale, is less expensive compared to molecular distillation equipment, as well as is much less expensive to operate. More importantly, the distillate fraction is as low as 1% compared to the 20% for molecular distillation.

Deodorization of oils by steam stripping is commonly the finishing step in edible oil processing, including those processes involving molecular distillation such as to reduce cholesterol. The typical equipment employed contains set stations which sparge steam into a body of oil with high baffeling. Conventional deodorization can remove some sterol materials, but such reduction is very modest. It is another surprising benefit of the present invention that the present process deodorizes the fish oil simultaneously with cholesterol level reduction without substantial destruction of desirable n-3 fatty acids. Thus, it is another surprising advantage of the present invention that the conventional oil finishing deodorization step is made optional rather than mandatory for the realization of clean fish oils. In certain embodiments of the present invention a deodorization step may still be wished to be practiced such as when cleaned fish oils of highest possible purity and quality are desired.

SUMMARY OF THE INVENTION

In its method aspect, the present invention

relates to physical processes for the deodorization and reduction in the cholesterol levels of a freshly refined or reverted fish oil.

5 The fish oil is first deaerated to an oxygen level of less than about 0.1% dissolved oxygen (by volume). The deaeration step involves subjecting the fish oil to a vacuum with or without sparging an inert gas through the oil.

10 Thereafter, about 1 to 15%, preferably 2 to 5% by weight of steam is added to the fish oil while under vacuum, 1-7 mmHg absolute and the mixture heated to about 400 to 550°F, preferably 430 to 500°F, most preferably 460 to 480°F.

15 The mixture is flash vaporized by introduction into a zone such as a disengaging or flash chamber having a pressure of about 1-7 mmHg, to form a loaded steam vapor phase and a par-treated fish oil phase.

20 The par-treated fish oil is thin-film stripped with 1 to 15%, preferably 2 to 5% by weight countercurrent steam at 1 to 7 mmHg, to provide a clean fish oil of reduced cholesterol content.

The cleaned fish oil is cooled under vacuum, optionally with sparge steam, then anaerobically stored.

25 In its product aspect, the present invention resides in new fish oil products essentially characterized in part by substantially complete removal of non-esterified cholesterol whereby the overall cholesterol levels are reduced from native levels by up to 65% or more. The novel fish oils are also characterized
30 by the bland flavor of clean fish oil but yet retain high levels of n-3 fatty acids.

The combined total n-3 fatty acid content, especially EPA plus DHA, is at least about 95% of the level of the starting material.

BRIEF DESCRIPTION OF THE DRAWINGS

Figures 1 and 1A are a schematic flow chart diagram of one embodiment of the present method.

DETAILED DESCRIPTION OF THE INVENTION

5 In its process aspect, the present invention relates to economical, physical processes for the simultaneous reduction of cholesterol level and deodorization of fish oil. In its product aspect, the present invention relates to clean fish oils of
10 reduced cholesterol levels which are bland in flavor and which are nonetheless high in EPA and DHA, but substantially free of residual organic solvents (chemical extractants). Importantly, the present invention includes, inter alia, the step of thin-film steam
15 stripping preferably with high steam consumption rates. Each of the process steps as well as product use are described in detail below.

Throughout the specification and claims, percentages and ratios are by weight and temperatures
20 in degrees Fahrenheit unless otherwise indicated.

Fish oil(s) have long been a staple of commerce in this country and their description as well as extraction and conventional processing and use are well described in "Fish Oils" edited by M. E. Stansby,
25 Avi Publishing Co., Inc., Westport, CT (1967). Especially useful for their therapeutic value herein are oils having a total n-3 fatty acid content of greater than about 20% such as menhaden oil, herring, capelin, anchovy, cod liver, salmon oil and sardine oil. The
30 up-stream processing of fish oils to produce the present starting materials is similar to that of vegetable oils. Typically, the processing can comprise conventional refining, bleaching, deodorization, and optionally winterizing and degumming. Conventional hydrogenation,
35 however, is to be avoided. Typically, however,

deodorization of an oil is the very last step in oil processing. The fish oil so prepared is referred to herein as "refined" fish oil and thus refers to undeodorized fish oil which has been refined, bleached, and optionally degummed and/or winterized.

Useful herein as the starting material are both freshly refined fish oil and reverted fish oil. By the term "reverted fish oil" is meant fish oil which has been previously refined and deodorized but which has subsequently undergone degradation by the passage of time and/or exposure to oxygen and thus has reverted back to an unpalatable state. Of course, other fats and oils with high non-esterified or free cholesterol levels can also be processed successfully including, by way of example, 1) dairy fats such as butter oil, 2) animal fats and lard, especially lard, and 3) marine fats. The treatment of these fats is also contemplated as within the scope of the present invention. It is to be appreciated that the present invention finds particular suitability for use in treating fish oil and throughout the specification other oils are contemplated even though fish oil is specifically described. However, minor changes in processing conditions may be necessary in order to achieve optimum results when specific other oils are being treated.

No attempt is made herein to distinguish between "fats" and "oils" as is sometimes made in the art since at the operating conditions of the present invention, the fatty triglyceride materials being processed are in a fluid state whether or not they exist in that physical form at room temperature.

The starting fish oil whether freshly refined, reverted or mixtures thereof is characterized by an initial or native cholesterol level. The exact cholesterol level varies depending upon such factors

as fish species, seasonality, geographical catch location and the like. Generally, however, such cholesterol levels can range from about 350 to 700 mg/100 gm of fish oil.

5 The starting fish oil is desirably first deaerated in conventional manner. (See for example, U.S. 3,506,696 issued Aug. 14, 1970 to Baker et al. and is incorporated herein by reference.) Deaerating the oil can be accomplished by subjecting the oil
10 to a partial vacuum and/or by sparging an inert gas, e.g., nitrogen through the oil. For example, initially, a pressure of about 25 mmHg is maintained for about 5 to 15 minutes. Thereafter, a modest nitrogen sparge can be continued for about 5 to 10 minutes. The
15 particular technique or conditions employed are not critical so long as after deaeration the total dissolved oxygen is less than about 0.1% by volume, preferably less than about 0.05%, prior to subsequent processing in order to prevent oxidation of the oil by dissolved
20 oxygen.

 Now referring to FIG. 1, there is shown an embodiment of the present process designated generally by reference numeral 10. A deaerated fish oil feed
11 is pumped by pump 23 so as to pass through a back
25 pressure valve 12 which maintains the entire system at a pressure of 1-7 mmHg which is provided by any conventional vacuum means 18 such as a steam jet system, e.g., four-stage. Optionally, the fish oil 11 may be modestly preheated such as with a first heating
30 means 13 such as a plate heat exchanger so as to provide a preheated fish oil 14. Then the deaerated fish oil 14 is, in a highly preferred embodiment, mixed under vacuum with a first steam feed 15, either wet or dry but preferably dry to form a steam/oil mixture
35 17. While cholesterol removal can be accomplished without using the first steam feed 15, better removal

of odoriferous constituents is achieved when steam is admixed with the oil feed and there is a corresponding improvement in stability of the finished fish oil.

The fish oil and steam mixture 17 can then be heated with a second indirect heat means 25 such as in a rising-film pre-evaporator using a heat transfer media 19 such as Dowtherm™ A to raise the temperature of the mixture 17 to essentially about 400 to 550°F, preferably 430 to 500°F, most preferably about 460 to 480°F. Such indirect heating is the preferred method for heating the steam/oil mixture 17 although other techniques, e.g., additional steam addition can also be used. Generally, steam consumption rates, if indirect heating is employed, run for this admixture 17 from about 0.1 to 15 lbs. of steam per 100 lbs. of fish oil, preferably about 1 to 5 lbs./100 lbs.

Conventional equipment can be used for such mixing and heating, such as simply sparging the steam 15 in line. The first steam feed's 15 condition or steam quality is not critical since thereafter the fish oil/steam mixture 17 is to be heated to the prescribed temperature range with the indirect heating being employed. Of course, the higher the steam enthalpy, the less indirect heating will be required.

Thereafter, the oil and steam mixture 17 passes optionally through means for controlling the oil feed rate 21 such as an ordinary throttling valve and is introduced to a zone or headspace 16 of relatively lower pressure to flash vaporize the steam and to flash off a portion of the cholesterol and odoriferous constituents in the oil to produce a loaded steam vapor 20 in the vapor headspace 16 and a par-treated fish oil 22. The loaded steam vapor 20 is pulled to a means for collecting the vapor 24 such as a conventional condenser or scrubber and a distillate 26 formed thereby collected for disposal and which

contains the odoriferous constituents and cholesterol in highly concentrated form.

The downwardly falling par-treated fish oil 22 is then essentially very thin-film steam stripped with a second, countercurrent steam feed 28, such as by using steam stripper 30. It is important during the practice of this step to maintain the pressure in the stripper 30 at about 1 to 7 mmHg, preferably about 2-3 mmHg. The temperature of the fish oil is essentially maintained at about 400 to 550°F, preferably 430 to 500°F, most preferably 450 to 470°F, such as by controlling the oil feed rate, and/or the temperature of the heat transfer media e.g., Dowtherm™ A or Therminol™ 66 and/or the amount of stripping steam 28. The stripping steam rate is maintained at 1 to 15%, preferably 2 to 5%.

Equipment useful for the practicing of both the flash vaporization step and the thin-film steam stripping step and thus combine both the vapor head 16 and stripper 30 are available commercially from Artisan Industries, Inc. The equipment and operation are described in U.S. Patent No's. 3,198,241 (issued Aug. 3, 1965 to J. L. Baird); 3,393,133 (issued July 16, 1968 to J. L. Baird) and 3,620,283 (issued Nov. 16, 1971 to Brown et al.) each of which are incorporated by reference. Generally, these patents are directed to apparatus and methods of fluid material treatment wherein liquid material to be stripped is cascaded over an alternating series of tubular jackets and discs, yielding a continual disruption of the film on the internal surface of the tube jacket in the vertical fall path after relatively short vertical distances and in which heat is supplied primarily to the continuous vertically moving, relatively thin liquid film phase. In this manner heat is conserved, hot and dry spots on the disc surface are prevented,

and the ease and efficiency of handling heat-sensitive materials is enhanced. The placing of the heat exchange fluid in an indirect heat exchange relationship primarily with the thin-film phase of the internal side of the tube permits the more efficient concentration and evaporation of materials.

The stripper 30 is commonly referred to as a tube and disc type evaporator. Generally, this type of equipment is used for different purposes, namely solvent recovery, e.g., hexane removal from soybean oil, and thus typical industrial focus has been given to optimization of the distillate fraction. It is highly surprising to select such equipment for use herein and such selection goes against the strongly held beliefs in the art with respect to the utility of such equipment for use as contemplated herein. Moreover, the equipment is used herein in an unconventional manner to achieve the present advantages. A first difference in using the equipment in the present invention is in the utilization of steam in admixture with the oil feed in the pre-evaporator, prior to the vapor head zone in preferred embodiments. Typically, oil and solvent mixtures are fed to the vapor head rather than steam and oil mixtures. Other differences are noted below in operating techniques.

The stripping steam feed 28 is preferably introduced at the bottom of the steam-stripper 30 so as to countercurrently contact the par-treated fish oil which is introduced into the top of the apparatus 30. The present thin-film steam stripping step is further importantly characterized by the steam consumption rate which can range from about 1 to 15, preferably about 2 to 5 lbs./100 lbs. of fish oil (0.01 to 0.15:1 steam to oil). Insufficient steam can undesirably lead to polymerization of the oil or at least inadequate stripping of free fatty acids and odoriferous compounds,

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thus yielding a product which is insufficiently deodorized. Similarly, reversion can occur more rapidly during storage if the undesirable compounds are not stripped away. Lastly, the cholesterol removal is much less efficient. Excessive steam consumption, however, can lead to much higher operating costs in the form of increased load on the vacuum system and further to incurring avoidable costs due to product loss through carry-over and unnecessary steam consumption. While high steam consumption adds to overall processing costs, steam costs are generally a much smaller cost compared to costs associated with product losses. Of course, the steam introduced to the bottom of the thin-film stripper is also pulled to the condensor or scrubber 24.

The rate at which the oil feed is fed to the thin-film stripping step is also an important independent process variable since the feed rate affects film thickness in the stripper 30. Another important distinguishing aspect of the present process is that the present process is practiced at much lower feed rates than is recommended by the equipment manufacturer. For example, for equipment rated with a throughput capacity of 25-100 lbs./hr., the presently employed feed rates typically range from about 5-8 lbs./hr. Importantly, the feed rates are controlled such that the film thickness ranges from about 1 to 25 mm and preferably about 1 to 5 mm in the thin-film stripper 30. Indeed, while the manufacturer describes the oil as falling in a thin-film, when viewed under the present, essential operating conditions, the oil/steam mixture appears to fall through the apparatus as a "rain" or mist or as very fine droplets. Thus, the present process is practiced at the far extreme end of thin-film stripping.

In addition to film thickness, it is also

important that the evaporative surface of the thin-film stripper is maintained at about 400 to 650°F, preferably about 425 to 600°F, and most preferably about 450 to 550°F using internal circulation of auxiliary heating media 32 through the stripper's 30 plumbing to provide further indirect heating of the oil for process control.

While not wishing to be bound by the present theory, it is speculated herein by virtue of the present unique thin-film technique, that the number of theoretical plates is dramatically increased and thus mass transfer rates increased so as to effect removal of the non-esterified cholesterol notwithstanding the similarity, broadly speaking, between the operating conditions and residence times of conventional steam stripping deodorization and the present method. Regardless of the theoretical mechanism, the present invention substantially reduces the overall or native cholesterol level in fish oil by up to two-thirds or more while conventional deodorization results in minimal cholesterol removal.

Most surprisingly, the fish oil is also effectively cleaned or deodorized simultaneously with the non-esterified cholesterol removal when processed under the conditions described above. Such a result is highly surprising to those familiar with oil processing and with the present equipment employed. The fish oil realized thus is also characterized by the clean color and flavor of a deodorized fish oil.

Exiting the stripper 30 is a clean oil stream 34, still at a pressure of 1-7 mmHg, which is then desirably cooled to room temperature in a conventional manner such as first passing through the plate heat exchanger 13 to conserve energy by preheating the oil feed 11 and finally with a cooling means 36 such as a cold water heat exchanger and anaerobically stored, e.g., with a positive pressure of an inert gas 38,

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preferably nitrogen. Sparge steam may also be optionally applied to the oil during cooling to preserve the highest quality possible. It is important to avoid any contact between fish oil at an elevated temperature and oxygen since oxidation rates are greatly accelerated with higher oil temperatures. Such contact between oxygen and other fats, e.g., beef tallow, is far less of a problem although nonetheless significant due to lesser degrees of polyunsaturation.

The described process is useful for realizing a finished fish oil 40 which is reduced in cholesterol, which is deodorized and which has a good flavor.

It is another advantage of the present invention that the residence time of the fish oil during processing is much reduced compared to conventional deodorization apparatus and techniques. Manufacturer's recommended processing conditions result in residence times of 15 minutes or higher. The present process, however, results in residence times of five minutes or less. Such reductions in residence times are highly beneficial since the n-3 fatty acids which are the beneficial constituents are also more susceptible to oxidation and reduced residence times minimizes their degradation.

In another preferred embodiment of the present invention depicted in FIG. 1A, however, a conventional deodorization step is added as a finishing step. Such an added deodorization step can be employed when the highest quality, cleanest flavor fish oil is desired, although extra costs are incurred. In this embodiment, the clean oil 40 is subjected to a "second" or conventional deodorization step using conventional deodorization equipment, methods and conditions. Oil deodorization is generally considered to be a fully developed art and one skilled in the art will have no difficulty selecting suitable apparatus and techniques from among those known. Generally, after

collecting in a surge/measuring tank 50, the oil 40
is fed to a conventional deodorizer 52. Steam 54
(280 to 360^oF, 50 to 150 psia) is introduced at the
bottom of each station and withdrawn from the vessel
5 and pulled to a scrubber. Supplemental heating 56
and cooling 58 can be employed. Column operating
pressures range from 1 to 7 mmHg by pulling a vacuum
from a convention vacuum source 60. The oil is not
refluxed. A twice deodorized fish oil product is
10 withdrawn from the deodorizer 52. Optionally filtered
in filter 64 and pumped to anaerobic storage.
Deodorization, however, typically results in some
fish oil loss, although such losses are modest, e.g.,
as low as 1%.

15 The clean fish oils realized by the present
invention are critically characterized in part by
removal of substantially all non-esterified cholesterol
and thereby also by an overall cholesterol level reduction
of at least 50% and even up to 65% or more from the
20 native or overall cholesterol level. While not wishing
to be bound by the proposed theory, it is speculated
herein that the unremoved or residual cholesterol
is more difficult to remove since it exists as cholesterol
esters which have higher molecular weights and thus
35 correspondingly lower volatilities. For lard, overall
cholesterol reductions of even as high as 90% and
higher have been obtained while in butterfat, reductions
of up to 95% have been obtained.

 In its product aspect, the present invention
30 contemplates a clean fish oil having a cholesterol
level of less than 50% of the native level, preferably
less than about 60-65% and wherein substantially all
(i.e., in excess of 98%) of the non-esterified cholesterol
has been removed. Qualitatively, the fish oil is
35 bland and substantially free of fishy flavor to the
average taster as measured by conventional sensory

evaluation of oils (see, for example, A.O.C.S. Recommended Practice, pgs. 2-83). In contrast to chemical methods of cholesterol removal from deodorized fish oil, the fish oil products of the present invention are substantially free of all organic solvents (i.e., less than 1 ppm). Further, unlike clean fish oil prepared from laboratory scale or molecular distillation, the present oils are importantly characterized by a light color and an absence of a strong chemical flavor.

When other oil feeds are employed, the present invention resides in clean, bland tasting oils characterized by the removal of substantially all non-esterified cholesterol and which nonetheless are free of chemical extractants.

The finished oils of the present invention as described have use as a food ingredient in a wide variety of food applications. In particular, the fish oil can be used to fortify other foods such as fabricated fish or battered and breaded fish portions. The novel fish oils can also be used in full or partial substitution for conventional oils in many food products such as salad dressing, mayonnaise and the like.

Example 1

A commercially obtained menhaden oil was used as the starting material having iodine value of 186, EPA content of 14.6%, DHA content of 11.2%, cholesterol level of 0.470%, and free fatty acid of 0.08%.

A semi-pilot plant oil processing system conforming substantially to Figure 1 was utilized to deodorize the above refined fish oil while simultaneously reducing its cholesterol level. The processing system employed a Model No. ESSJ 13-2 Evaporator, steam stripper (available from Artisan Industries, Inc.) as the thin film steam stripping

column 30. The processing system was started up by drawing a vacuum on the entire system, heating the pre-evaporator 25 and thin film steam stripping sections 30 using Dowtherm™ HT, (partially hydrogenated terphenyl) as the heat transfer media, establishing steady state conditions with the stripping steam 15 and 28 in the pre-evaporator 25 and steam stripper sections 30, respectively and charging the system 10 with a continuous feed stream of refined fish oil.

The refined fish oil was first deaerated in a feed tank using a vacuum of 25 mmHg absolute pressure and a subsequent nitrogen sparge. Next the feed was pumped through a back pressure valve 12 at a rate of 5.2 lb/hour (2.36 kg/hr) into the processing system, maintained at 1.5 mmHg pressure, where 0.75 lb/hour (0.34 kg/hr) dry steam 15 (14.4% steam:oil ratio) was sparged in line to form a steam/oil mixture. The mixture was heated in the rising film pre-evaporator section 25, which had a 1.96 Ft^2 (0.18 m^2) heating surface area and 550°F Dowtherm™ HT circulating in the jacket as heat transfer media 19. The steam/oil mixture achieved a final temperature of about $460\text{--}480^\circ\text{F}$ at the top of the pre-evaporator 25, while still at 1.5 mmHg pressure.

Next the fish oil/steam mixture flowed into the disengaging chamber or vapor head 16, where the vapor phase was flash vaporized and separated from the liquid phase. The loaded vapor phase 20, consisting primarily of steam with free fatty acids, organic compounds, cholesterol, etc., was drawn to the vacuum system and collected as waste. The liquid phase 22, consisting of par-treated fish oil, was then thin film countercurrent steam stripped in the stripping section 30 which consisted of 13 tube and disc stages which were heated using the Dowtherm™ HT heat transfer media 32. The oil cascaded over the stages by gravity

in a very fine film of a thickness visually measured of about 10 mm, which however in the final stages broke down into a mist of droplets with diameter of 0.2-5 mm. Simultaneously, 0.75 lb/hour (0.34 kg/hr) dry steam (14.4% steam:oil ratio) 28 was injected at the bottom of the column 30 to countercurrently strip the volatile components from the fish oil, such as free fatty acids, cholesterol, etc.

After steam stripping, the oil was immediately cooled to ambient temperature, while still under vacuum, using a small water cooled heat exchanger 36. The fish oil was then collected in nitrogen flushed containers for storage.

The final fish oil product had iodine value 184, EPA content of 14.4%, DHA content of 10.8%, cholesterol level of 0.144%, and free fatty acid of 0.03%. The fish oil processed as per the above had the bland flavor of deodorized edible oil, while maintaining the EPA and DHA omega-3 fatty acid content yet nonetheless was characterized by a significant 69.4% cholesterol level reduction.

Example 2

A commercially available Menhaden oil was used as the starting material having iodine value of 186, EPA content of 13.0%, DHA content of 9.9%, cholesterol level of 0.463%, and free fatty acid of 0.23%.

A semi-pilot plant apparatus conforming to Figure 1 and as used in Example 1 was utilized to deodorize the above refined fish oil while simultaneously reducing its cholesterol level. The process was started up substantially as described in Example 1 by drawing a vacuum on the entire system, heating the pre-evaporator 25 and steam stripper 30 using Dowtherm™ HT, establishing steady state conditions with the stripping steam 28 in the steam stripper

30 (with none in the evaporator), and charging the system with a continuous feed stream of refined fish oil.

The refined fish oil was first deaerated in a feed tank using a vacuum of 25 mmHg absolute pressure and a subsequent nitrogen flush. Next the feed was pumped through a back pressure valve 12 at a rate of 8.10 lb/hour (3.68 kg/hr) into the processing system, maintained at 2.5 mmHg pressure. No steam was added to the fish oil prior to heating in the pre-evaporator 25 (heating area 1.96 Ft² or 0.18 m²) as the fish oil was heated to a final temperature of 480-500°F at the top of the evaporator using the Dowtherm™ HT as the media 19 in the jacket.

Next the fish oil passed into and through the vapor head 16 to flash vaporize into a liquid fraction and a vapor fraction. The liquid fraction passed into the stripping section 30 while still at 2.5 mmHg pressure. The par-treated fish oil cascaded over the 13 tube and disc stages, which were heated with 550°F Dowtherm™ HT as media 32, in a very fine thin film of a thickness measured at about 10 mm which however in the final stages broke down into a mist of small droplets with diameters of 0.2-5 mm. Simultaneously, 0.75 lb/hour (0.34 kg/hr) dry steam (9.26% steam:oil ratio) 28 was injected at the bottom of the column 30 to countercurrently strip the volatile components from the liquid phase fish oil 22, such as free fatty acids, cholesterol, etc. The vapor phase 20 produced by the flash vaporizing containing the stripping steam and these volatile components, was separated from the liquid phase in the vapor head 16 and drawn to the vacuum system 24, where it was collected as waste.

After the oil was thin film steam stripped, it was immediately cooled to ambient temperature,

while still under vacuum, using a small water cooled heat exchanger 36. The fish oil 40 was then collected in nitrogen 38 flushed containers for storage.

The final fish oil product had iodine value 182, EPA content of 12.9%, DHA content of 9.6%, cholesterol level of 0.137%, and free fatty acid of 0.04%. The fish oil processed as per the above had the bland flavor of deodorized edible oil, while maintaining the EPA and DHA omega-3 fatty acid content and had a significant 70.4% reduction in cholesterol level without any steam in the pre-evaporator section.

Example 3

Anhydrous butterfat was commercially obtained from Mid America Farms to serve as starting material, having cholesterol level of 0.208% and a free fatty acid level of 0.41%.

A semi-pilot plant apparatus conforming to Figure 1 and as used in Examples 1 and 2 was utilized to reduce the cholesterol content of the above butterfat. The process was started up by drawing a vacuum on the entire system, heating the pre-evaporator 25 and steam stripper 30 using Dowtherm™ HT as media 19 and 32, establishing steady state conditions with the stripping steam 15 and 28 in the pre-evaporator and steam stripper 30, and charging the system with a continuous feed stream of anhydrous butterfat.

The butterfat was first deaerated and preheated to 120°F in a feed tank using a vacuum of 25 mmHg absolute pressure and a subsequent nitrogen sparge. Next the butterfat feed 14 was pumped through a back pressure valve 12 at a rate of 7.4 lb/hour (3.36 kg/hr) into the processing system 10, maintained at 1.5 mmHg pressure, where 0.720 lb/hour (0.33 kg/hr) dry steam 15 (9.7% steam:fat ratio) was sparged in line to form a steam/fat mixture. The mixture 17 was heated in the rising film pre-evaporator 25, which had 1.96

Ft² heating surface area and 550²¹°F Dowtherm™ HT circulating in the jacket as media 19. The mixture achieved a final temperature of 470-490° F. at the top of the pre-evaporator 25, while at 1.5 mmHg absolute pressure.

Next the preheated or par-treated butterfat/steam mixture flowed into the disengaging chamber or vapor head 16, for flash vaporizing, where the liquid phase was separated from the vapor phase. The vapor phase 20, consisting primarily of steam with free fatty acids, cholesterol, color bodies, and other volatiles, was drawn to the vacuum system 24 and collected as waste. The liquid phase 22, consisting of par-treated butterfat, was thin film countercurrent steam stripped in the steam stripper 30 consisting of 13 tube and disc stages which were heated using Dowtherm™ HT. The fat cascaded over the stages by gravity in a very fine film, which broke down in the last stages into a mist of droplets with diameter of 0.2-5 mm. Simultaneously, 0.750 lb/hour (0.33 kg/hr) dry steam 28 (10.1% steam:fat ratio) was injected at the bottom of the stripper column 30 to countercurrently strip the volatile components from the butterfat, such as free fatty acids, cholesterol, color bodies, etc.

After the fat was thin film steam stripped, it was immediately cooled to 130° F while still under vacuum, using a small water cooled heat exchanger 36. The butterfat was then collected in nitrogen flushed containers for anaerobic storage.

The final butterfat product had cholesterol level 0.010% and free fatty acid of 0.01%. The butterfat processed as per the above had the bland flavor and functionality of commercial butterfat, losing only some color bodies, with a significant 95% reduction in cholesterol level.

WHAT IS CLAIMED IS:

1. A method for deodorizing and reducing cholesterol level of fats and oils comprising the steps of:
 - 5 A. providing a feed stream of refined or reverted, deaerated oil having less than about 0.1% by volume of dissolved oxygen, said oil having a native cholesterol and odoriferous material level;
 - 10 B. introducing and intimately mixing a first steam feed under vacuum into and with the deaerated oil to form a steam and heated oil mixture;
 - C. heating the mixture to a temperature of about 400 to 550°F;
 - 15 D. flash vaporizing the mixture to vaporize the steam and at least a portion of the cholesterol and odoriferous materials to realize a par-treated oil liquid phase and loaded vaporous steam phase and wherein the temperature is maintained at about 400 to 550°F;
 - 20 E. thin-film steam stripping the par-treated oil in a stripper having an evaporative surface at a pressure of about 1 to 7 mmHg. with a second countercurrent steam feed to strip a portion of the cholesterol and odoriferous material to provide a clean oil having a
25 cholesterol content less than its native level and wherein the second steam feed is admixed with the loaded vaporous steam phase;
 - F. removing the loaded vaporous steam phase;
 - 30 G. cooling the clean oil under vacuum to a temperature of less than about 100°F; and
 - H. anaerobically storing the cleaned oil.
2. The process of Claim 1 wherein in step E the clean
35 oil has a cholesterol level of less than about one-half the native cholesterol level.

3. The process of Claim 2 wherein in step E, the oil to steam ratio ranges from about 1:0.01 to 0.15.
4. The process of Claim 3 wherein the oil is selected from the group consisting of fish oil, animal fats, marine fats, dairy fats and mixtures thereof.
5. The process of Claim 4 wherein the feed stream is deaerated to about 0.05% by volume of oxygen, and wherein the thin-film steam stripping is practiced in a tube and disc evaporator, and wherein the thin-film steam stripping is practiced so as to have a film thickness on the evaporative surface ranging from about 1 to 25 mm.
6. The process of Claim 5 wherein the weight ratio of first steam feed to oil ranges from about 0.001 to 0.15:1.
7. The process of Claim 6 wherein the thin-film steam stripping is practiced so as to have a film thickness ranging from about 1 to 5 mm.
8. The process of Claim 7 wherein in step E, the oil to steam ratio ranges from about 1:0.02 to 0.05.
9. The process of Claim 8 wherein in step C, the mixture is heated to a temperature ranging from about 430 to 500°F.
10. The process of Claim 9 wherein in step E, the clean oil has a cholesterol level of less than about 35% of the native cholesterol level.
11. The process of Claim 10 wherein the evaporative surface is maintained at a temperature of about 400 to 650°F and wherein the loaded vaporous steam phase is removed by condensing the phase in a condensor.
12. The process of Claim 11 additionally comprising the step of:
deodorizing the clean oil.

13. The process of Claim 12 wherein step G is practiced with a steam sparge in a weight ratio of oil to steam ranging from about 1:0.01 to 0.15.
- 5 14. The process of Claim 13 wherein the oil is a fish oil.
15. The process of Claim 14 wherein in step C, the mixture is heated to a temperature ranging from about 460 to 480°F.
- 10 16. The process of Claim 12 or 14 wherein the oil comprises Menhaden oil.
17. The process of Claim 11 wherein the oil is a dairy fat.
18. The process of Claim 12 wherein the oil is a dairy fat.
- 15 19. The process of Claim 11 or 12 wherein the oil is an animal fat.
20. A food product comprising a fat substantially free of non-esterified cholesterol and chemical extractants and wherein the fat is selected from
20 the group consisting of fish oil, animal oil, marine oil, dairy fats and mixtures thereof.
21. The food product of Claim 20 wherein the fat is fish oil.
- 25 22. The food product of Claim 21 wherein the fat has an n-3 fatty acid content of about 20% by weight.
23. The food product of Claim 22 wherein said fish oil is Menhaden oil having a total n-3 fatty acid content of greater than about 20%.
- 30 24. The food product of Claim 20, 21, 22 or 23 wherein the fat comprises about 0.1% to about 50% by weight of the product.

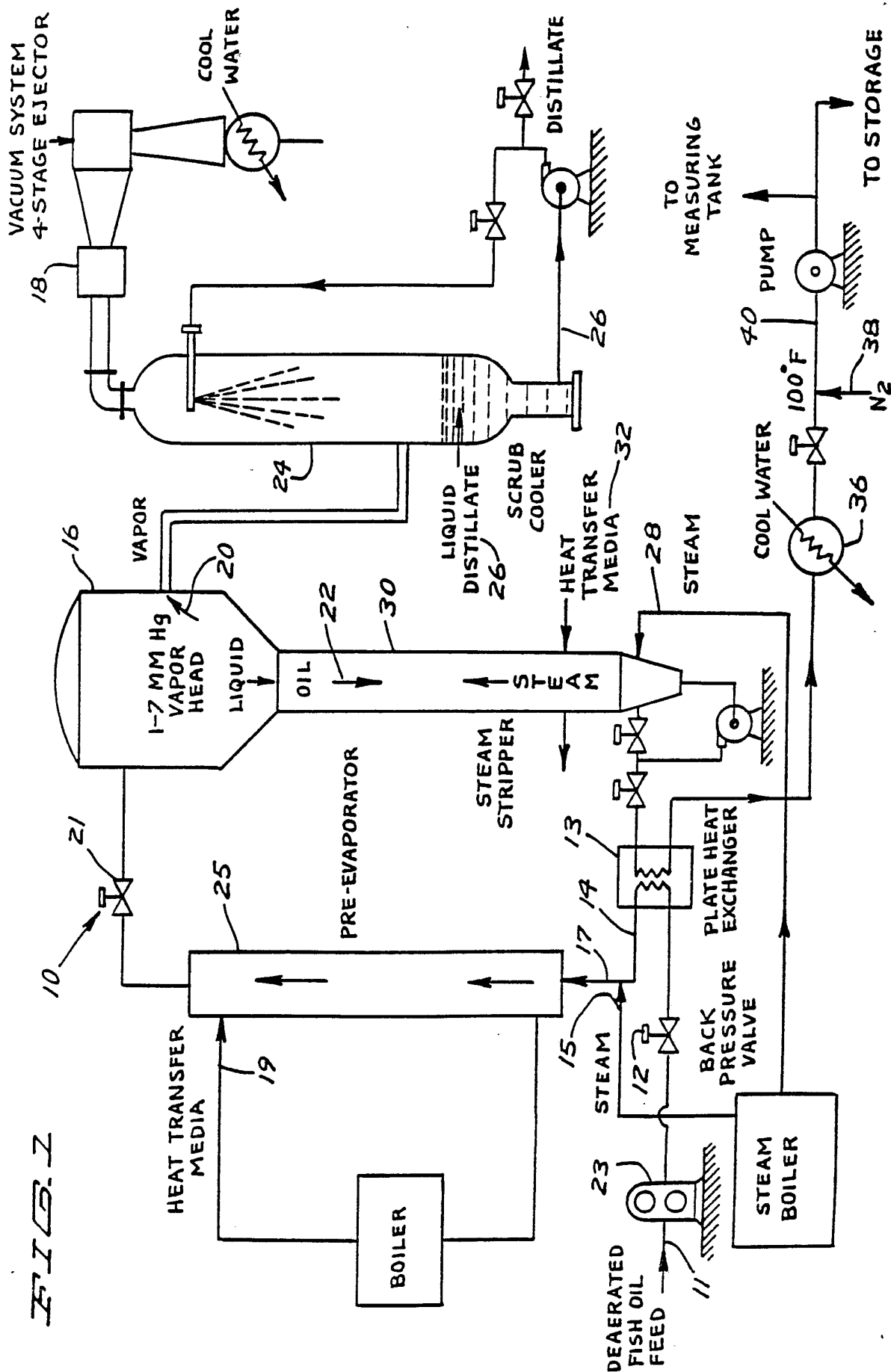
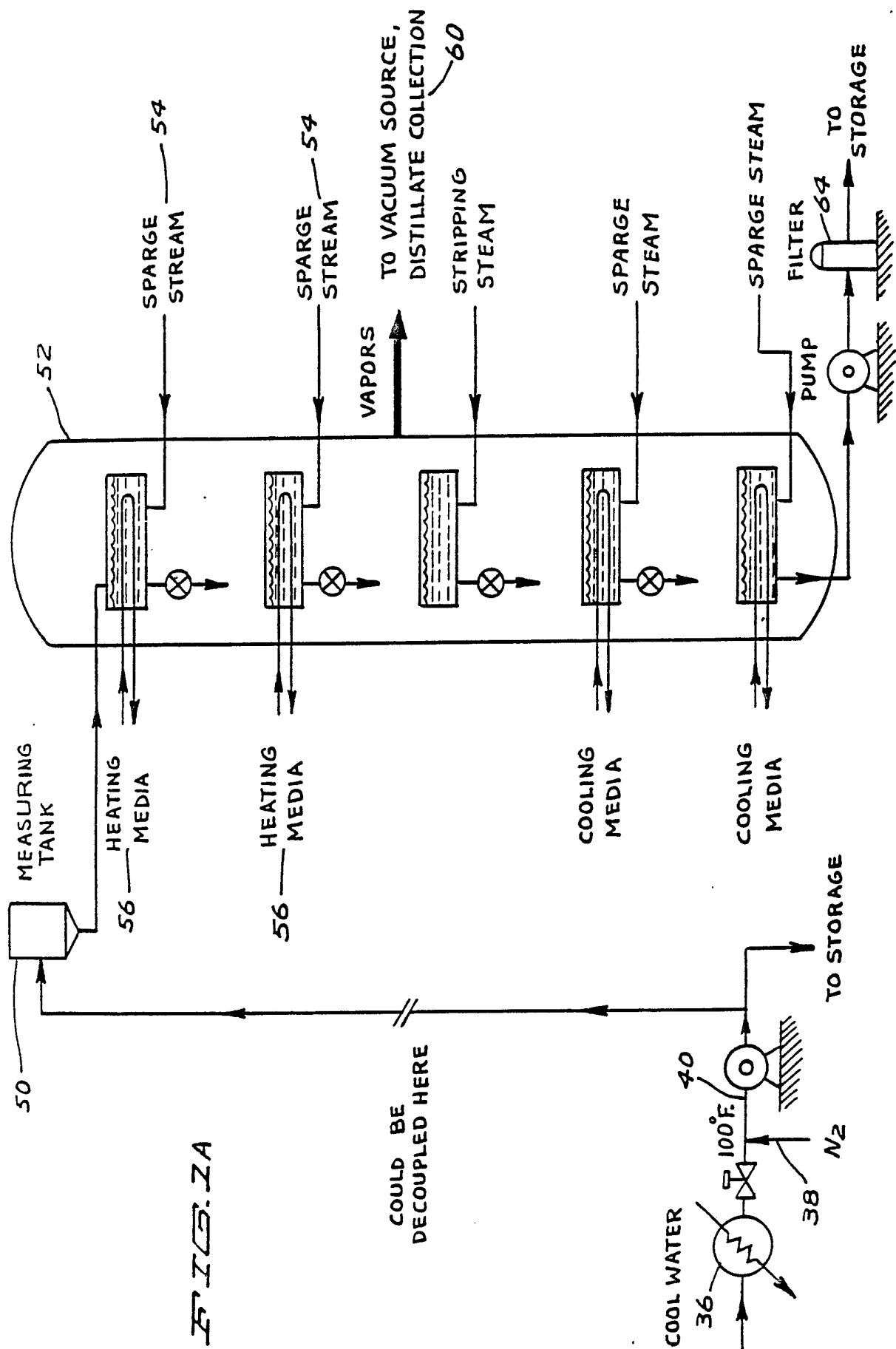



FIG. 1



INTERNATIONAL SEARCH REPORT

International Application No. **PCT/US87/02738**

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) ¹		
According to International Patent Classification (IPC) or to both National Classification and IPC IPC (4): A23D 5/00; C11B 3/00 U.S. CL.: 426/417; 260/420		
II. FIELDS SEARCHED		
Minimum Documentation Searched ⁴		
Classification System	Classification Symbols	
U.S.	426/417, 601, 607 260/420, 428, 428.5	
Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched ⁵		
III. DOCUMENTS CONSIDERED TO BE RELEVANT ¹⁴		
Category ⁶	Citation of Document, ¹⁶ with indication, where appropriate, of the relevant passages ¹⁷	Relevant to Claim No. ¹⁸
A	US, A, 2,804,427 (SURIANO) 27 AUGUST 1957 See claim 7.	1-19
A	US, A, 3,506,696 (BAKER ET AL) 14 APRIL 1970 See column 2, lines 9-35.	1-19
X	US, A, 4,093,540 (SEN GUPTA) 06 JUNE 1978, See abstract, lines 12-15, column 2, lines 6-16, and column 6, lines 37-39.	20-24
<div style="display: flex; justify-content: space-between;"> <div style="width: 45%;"> <p>¹⁵ * Special categories of cited documents:</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> </div> <div style="width: 45%;"> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</p> <p>"&" document member of the same patent family</p> </div> </div>		
IV. CERTIFICATION		
Date of the Actual Completion of the International Search ²	Date of Mailing of this International Search Report ³	
10 NOVEMBER 1987	26 JAN 1988	
International Searching Authority ¹	Signature of Authorized Officer ¹⁰	
ISA/US	 C. CALLAHAN	

FURTHER INFORMATION CONTINUED FROM THE SECOND SHEET

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☐ V. OBSERVATIONS WHERE CERTAIN CLAIMS WERE FOUND UNSEARCHABLE 10

This international search report has not been established in respect of certain claims under Article 17(2) (a) for the following reasons:

1. ☐ Claim numbers _____, because they relate to subject matter 12 not required to be searched by this Authority, namely:

2. ☐ Claim numbers _____, because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out 13, specifically:

☒ VI. OBSERVATIONS WHERE UNITY OF INVENTION IS LACKING 11

This International Searching Authority found multiple inventions in this international application as follows:

- I. Claims 1-19, drawn to a method for deodorizing and reducing cholesterol level of fats and oils; Class 260, subclass 420.
- II. Claims 20-24, drawn to a fat-containing food product;
- Class 426, subclass 6014.
1. ☒ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims of the international application. Telephone Practice
2. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims of the international application for which fees were paid, specifically claims:

3. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claim numbers:
4. ☐ As all searchable claims could be searched without effort justifying an additional fee, the international Searching Authority did not invite payment of any additional fee.

Remark on Protest

- ☐ The additional search fees were accompanied by applicant's protest.
- ☐ No protest accompanied the payment of additional search fees.