

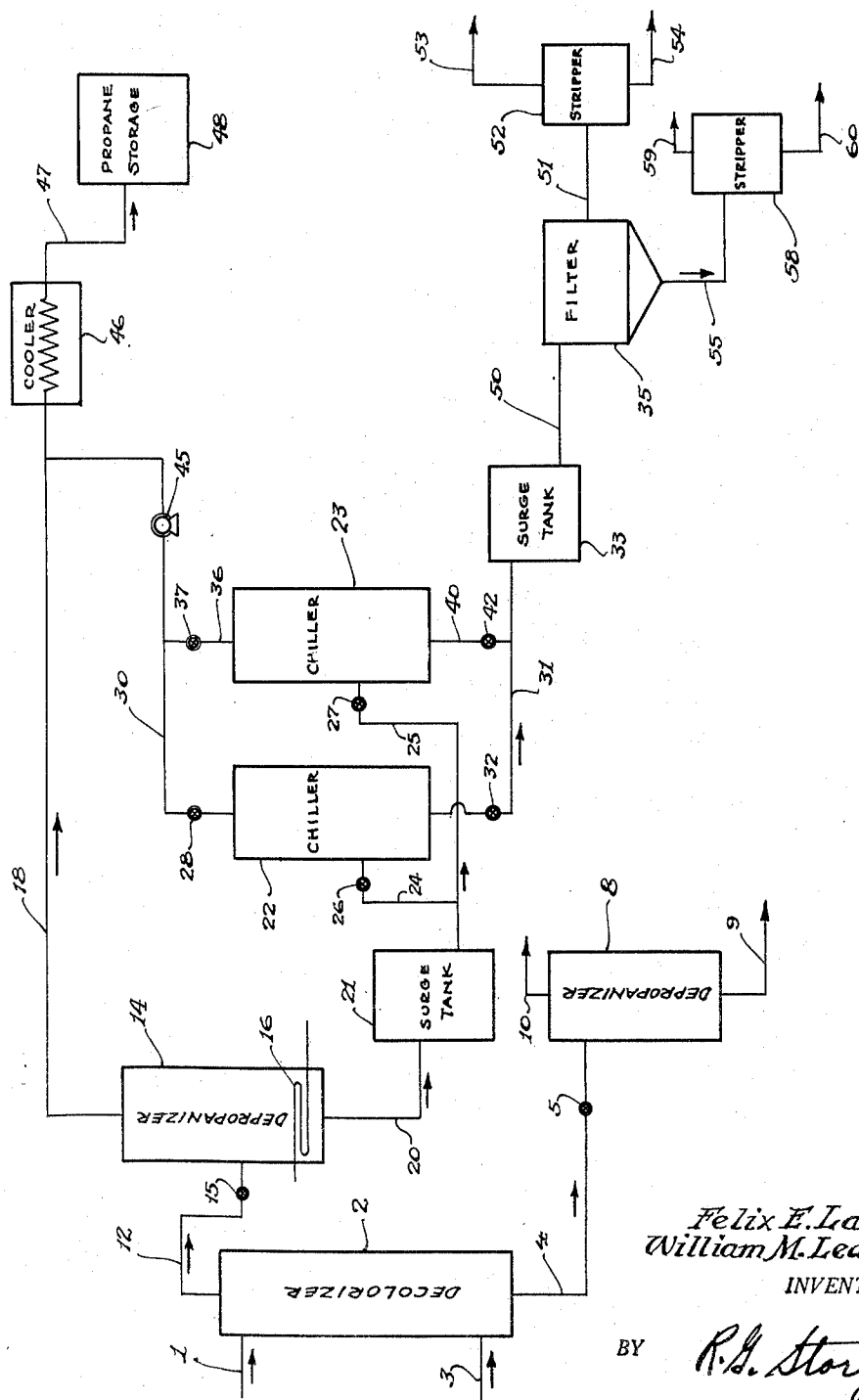
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SOLVENT FRACTIONATION OF FATTY MATERIALS

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SOLVENT FRACTIONATION OF FATTY MATERIALS

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This invention relates to the treatment of fatty materials to separate desired constituents. More particularly, the invention has to do with the solvent fractionation of fatty materials such as fats, fatty oils and fatty acids to produce purified fractions of different solubility. The invention has particular application to the treatment of relatively crude materials to produce fractions thereof improved in color and more adapted for specific commercial uses.

Natural fatty materials often contain undesirable color bodies which are necessary to be removed in the manufacture of commercial products. These color bodies are intensified by normal processes of oxidative deterioration and in many cases render the fatty material unsuited for commercial usage. The color bodies are difficult to remove and often require expensive factory treatment resulting in low yields. Many fatty materials are composed of relatively high melting point and relatively low melting point mixtures. The low melting point material is in greater demand than the higher melting point material which is of limited commercial use as now prepared.

An object of the present invention is to provide an improved method of removing the color from fatty materials.

Another object of the invention is to provide a process for treatment of fatty materials heretofore unavailable for the production of high grade products.

Another object of the invention is to remove certain impurities from fatty materials which interfere with subsequent processing operations.

A further object of the invention is to provide an improved process of treating fats and fatty oils whereby increased yields of desired products are obtained.

A still further object of the invention is to produce from crude fatty materials a higher melting point fraction of enhanced value.

Also an object of the invention is to provide a more efficient process for improving color and separating fractions of fatty materials.

The fatty materials which may be treated according to the present invention comprise all grades of tallow and greases including edible and inedible varieties, all types of vegetable and marine oils whether hydrogenated or unhydrogenated, and fatty acids derived from animal, vegetable or marine origin as well as synthetic esters and derivatives of the aforesaid fatty acids.

Heretofore in the manufacture of fatty oils from greases a selection of raw material was made

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to provide sufficiently high quality of products. The material then required acid washing to remove impurities such as emulsifying agents and crystallizing inhibitors. The product was then slowly crystallized by gradual reduction in temperature over an extended period of time. The crystallized product was then separated by pressing into an oil fraction and a solid stearin fraction. The process is complicated, inefficient, very laborious, and fails to produce sufficiently well separated fractions for most effective applications. The yields have been relatively low and the quality of the products is inferior.

Sperm oils have been treated heretofore in much the same manner as greases. The same disadvantages are in general inherent in the process of the treatment of marine oils as in the treatment of greases.

In the prior methods of manufacturing fatty acids such as red oil and stearic acid raw materials which are usually quite dark in color are often used. The resulting crude fatty acids are distilled to recover a light color distillate and to separate the color bodies as bottoms. The distilled fatty acids are then subjected to repeated crystallization and pressing to separate the saturated from the unsaturated fatty acids. In such an operation there are high losses in the distillation step due to polymerization sometimes amounting to as much as 12%. Also the seeding or crystallizing and pressing operations are expensive, require a large amount of equipment and require much hand labor.

In the treatment of foots resulting from the refining of vegetable oils such as cotton and soybean oil it has been customary heretofore to acidulate and distill the resulting released fatty acids. The losses in this operation are very high often running to as much as 25% to 35%. These products have been suitable only for use requiring a relatively cheap source of raw material.

The present invention contemplates broadly the treatment of the raw material with a normally gaseous hydrocarbon solvent to remove undesirable color bodies under relatively high temperature and pressure conditions and the subsequent treatment of the decolorized product under lower temperature and pressure conditions to separate the product into fractions of different solubility and having different degrees of saturation and melting point. The process further contemplates a correlation of the temperature and pressure conditions and a coaction of steps in the decolorization and solvent fractionation operations whereby minimum refrigeration com-

pression are required and maximum utilization of the sensible energy is secured. The invention also provides a continuous operation requiring a minimum amount of labor, equipment, facilities and solvent handling.

In accordance with the present invention the fatty material to be treated is subjected to a decolorizing operation in the presence of a normally gaseous hydrocarbon such as propane at temperatures near the critical point of the hydrocarbon and under sufficient pressure to maintain the hydrocarbon in liquid phase. In the decolorizing operation two phases are formed, the heavier phase comprising a solution of the color bodies and a lighter phase comprising a solution of the decolorized product. The heavier solution, or foots, is separated and the propane recovered leaving a dark viscous material similar to pitch. The solution of decolorized oil is subjected to a depropanizing operation under a reduced pressure. In the depropanizing operation the major portion of the propane is evaporated and the oil chilled to a substantially lower temperature. The remainder of the solution containing unvaporized propane is subjected to additional vaporization under a further reduced pressure. In the latter vaporization operation the oil is chilled to a sufficiently low temperature to crystallize the high melting point constituents, sufficient propane remaining in the product to enable the mixture to be filtered. The chilled product is then filtered to remove the crystalline portion from the solution of oil and propane. The higher melting point fraction and the oil fraction are subjected to solvent recovery operations to recover the propane. The propane volatilized in the depropanizing, chilling and recovery operations may be cooled and recompressed to recover the propane in a liquid form.

The conditions in the decolorizing operation may vary considerably. We have found that temperatures of 170° F. to 185° F. are satisfactory and preferably around 175° F. The pressure should be sufficient to maintain the hydrocarbon in a liquid phase at the temperature specified. Pressures within the range of 400 pounds to 530 pounds are satisfactory, and preferably around 465 pounds per square inch absolute. The ratio of propane to oil in the decolorizing operation may range from between 15 to 1 to 30 to 1 and preferably about 20 to 1 by volume. Ordinarily the proportions of the two phases in the decolorizing zone are about 40 of the decolorized solution to 1 of the foots although these may vary considerably depending on the temperature conditions. In general it is desirable from a yield and an efficiency standpoint to operate at as low a temperature as possible and yet maintain two phases.

The depropanizing operation may be carried out under a pressure of about 200 pounds to 300 pounds per square inch preferably about 250 pounds per square inch, and at a temperature of 100° F. to 135° F., preferably about 120° F. Under these conditions sufficient propane is vaporized to reduce the ratio of propane to oil to around 4 or 5 to 1 and preferably about 4½ to 1. The vaporization of such quantity of propane would often cause undue cooling of the unvaporized solution for example it might be sufficient to reduce the temperature to around -40° F. to -50° F. Such low temperatures are undesirable and accordingly it is advantageous to add heat whereby the temperature is maintained at around 115° F. to 130° F. and preferably around 120° F. It is desirable to

maintain such temperature in the first stage of depropanization so that the further cooling and crystallization of the higher melting point fats can be brought about subsequently under controlled conditions which favor the growth of more readily filtered solids. Another advantage of maintaining such temperature in the first stage of depropanization is the fact that the pressure of the vapor formed is sufficient to permit liquefaction by a simple condensing operation which eliminates a great deal of mechanical compression. The heat may be supplied by any suitable means such as a direct heat exchange from a steam or electrical coil, or indirect heating by an electrical heater or steam jacket.

In the chilling operation the propane is slowly evaporated by a gradual reduction in the pressure, the pressure being reduced by this operation from about 250 pounds to around 30 to 75 and preferably around 50 pounds. The reduction in pressure will cause sufficient propane to be vaporized to reduce the ratio of propane to oil to about 3½ or 4 to 1. The evaporation of the propane causes a reduction in temperature of the remaining solution to around -20° F. to +40° F., preferably between 0° F. and +10° F. This chilling operation must be controlled so that the temperature drop is between 1 and 2° per minute, making the total chilling time between 1 and 2 hours depending upon the material being treated. A suitable crystal structure for good filtration is formed with a cooling speed of about 1¼° per minute, the optimum filtration range being obtained with approximately a 90 minute chilling cycle. The chilled solution may be filtered in any suitable type of pressure filter, preferably a continuous rotary filter enclosed within a vapor type shell. A suitable filter is one shown in the United States patent to Van Horn No. 2,050,007.

The invention will be more clearly understood from the following description read in connection with the accompanying drawing.

Referring to the drawing the fatty charging stock is introduced under pressure through the line 1 into the upper portion of a decolorizing tower 2. The tower 2 is preferably packed with a surface contact material such as Raschig rings not shown. The normally gaseous hydrocarbon solvent is introduced into the lower portion of the tower through the line 3. The fatty material being heavier than the propane begins to descend through the tower counter-current to the rising propane solution. The temperature at the top of the tower is preferably maintained 2 to 10° higher than the temperature at the base of the tower. The temperature conditions maintained in the tower facilitates the continuous separation of color bodies from the rising propane oil solution and enhances the efficiency of the operation from the standpoint of oil yield. Under the temperature and pressure conditions maintained in the tower a separation of phases occurs. The interface is near the bottom, preferably below the line 3 where the propane is introduced. The heavier phase in the lower portion of the tower is drawn off through the line 4 controlled by a valve 5 to a depropanizer 8. A reduced pressure is maintained in the depropanizer 8 by releasing the pressure through the valve 5. Any propane not spontaneously evaporated by reduction in pressure may be stripped out with steam. The depropanized oil or foots is withdrawn from the bottom of the depropanizer through the line 9. The propane vapors are withdrawn from the top of the depropanizer through the vapor line 10.

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While one depropanizing vessel is shown it is preferred to use three depropanizers so as to obtain a three stage depropanization. A suitable manner of operation of the depropanizers is to use 50 pounds in the first stage, 5 pounds in the second and steam stripping at atmospheric in the final stage. When three stages of depropanization are used the foots would be drawn off from the final steam stripping stage and the propane vapors would be withdrawn from each stage. The vapors from the first two stages of the depropanization would be pure propane whereas that from the final stage would be a mixture of propane and steam. The propane may be recovered from the vapors and reused in the system.

The solution of decolorized oil and propane is withdrawn from the top of the decolorizer through the line 12 to a depropanizer 14. The pressure in the depropanizer 14 is maintained substantially below that in the decolorizer 2 by operation of the reducing valve 15. The reduction in pressure will cause a vaporization of the major portion of the propane and a reduction in temperature of the unvaporized solution. In order to avoid excessive cooling of the solution in the depropanizer heat is applied thereto. In the drawing such heat is supplied by a steam coil 16 but other means of heating may be used. The propane vapors are withdrawn from the top of the depropanizer through the line 18 for recovery as explained hereinafter. The unvaporized solution is withdrawn from the lower portion of the depropanizer through the line 20 and charged to a surge tank 21. The surge tank 21 acts merely as a reservoir for the chilling vessels 22 and 23. The solution in the surge tank may be charged alternately to chillers 22 or 23 through the lines 24 or 25 controlled by valves 26 and 27.

In one method of operation a batch of oil is charged from the surge tank into one of the chillers such as vessel 22. The charge to the chiller is preferably insufficient to completely fill the chiller so as to provide a vapor space. After the vessel is charged the pressure is gradually released by the valve 23 in the vapor line 30. The reduction in pressure causes vaporization of the propane and a chilling of the oil. When sufficient vaporization and chilling has occurred the vapor line 30 is closed by valve 28 and the chilled oil is withdrawn from the bottom of the chiller through the liquid line 31 controlled by valve 32. The liquid line communicates with a surge tank 33 whereby the oil treated in the chiller may be withdrawn to the surge tank. The function of the surge tank 33 is to supply a steady stream of chilled solution to the rotary filter 35 which operates continuously. During the operation of the chiller 22 the chiller 23 may be charged with another batch of oil from the surge tank 21. When the charge in chiller 22 is sufficiently chilled and the valve 28 is closed the chilling operation may be commenced in the vessel 23. This chilling operation is substantially the same as that described in connection with vessel 22 and may take place during the discharge of the chilled product from the vessel 22. Vapors from chiller 23 are passed through vapor line 36 in which is located valve 37 to vapor line 30. The chilled solution in chiller 23 is conducted through a liquid line 40 controlled by valve 42 to the liquid line 31.

It will be observed that the operations of vessels 22 and 23 are alternate batch operations and are operated in such a manner as to give ade-

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quate time for the chilling and the formation of crystals. The charging and discharging times required for the loading and unloading of vessels 22 and 23 is small in comparison to the time consumed in the chilling operations so that the two vessels operating alternately maintain a steady supply of chilled solution for the continuous operation of the filter and use up sufficient warm solution from the surge tank 21 to allow continuous operation prior to the batch chilling. The vapors from the chillers 22 and 23 collecting in the vapor line 30 are compressed by the pump 45 and introduced into the vapor line 18. The combined vapors in the line 18 are chilled in a cooler 46 to a sufficiently low temperature to cause the vapors to liquefy. The liquid is passed through the line 47 to a propane storage tank 48.

The chilled oil from the surge tank 33 is conducted through the line 50 to a filter 35. The surge tank is preferably elevated somewhat above the filter 35 so that the oil feeds through the line 50 by gravity. Separation in filter 35 takes place under a positive pressure of propane gas, the solution passing through the rotary drum. The crystalline material is collected on the drum and subjected to a wash of cold propane to further reduce the liquid portion absorbed on the crystal surfaces. The crystal cake is removed from the rotary drum by a scraping mechanism which causes the crystalline material to fall into a trough where the temperature is increased sufficiently to cause a solution of the crystal fat in the propane present. The solution is then conducted out of the filter through line 51 to a solvent recovery system wherein the solvent is stripped of the fat, the solvent being withdrawn through the line 53 and the fat through the line 54. The liquid portion separated on the filter is combined with the wash solution and the mixture conducted through line 55 to a solvent recovery system 58, wherein the solvent is stripped out and solvent as vapors passed through the vapor line 59. The oil is withdrawn through the line 60.

As an example of the operation of the invention, a sample of brown grease was decolorized in 30 volumes of propane at a temperature of about 175° F. and under a pressure of about 475 pounds in a tower. A temperature gradient of about 3° F. between the top and bottom of the tower was maintained. The lighter decolorized liquid phase was withdrawn from the top of the tower and was introduced into a depropanizer with a reduction in pressure to about 250 pounds. The reduction in pressure caused sufficient propane to volatilize to reduce the propane to oil ratio to about 4½ to 1 by volume. In the latter operation additional heat was supplied by a low pressure steam coil to maintain the temperature of the product at about 118° F. The product was then introduced into a chiller wherein the pressure was gradually reduced to 40 pounds per square inch over a period of about 90 minutes. The gradual reduction in pressure was accomplished by boiling off about ½ volume of propane producing a temperature of about 10° F. and leaving a final ratio of propane to oil of about 4 to 1. The product was removed from the chiller, filtered, and the oil and stearin fractions stripped of their propane content. The yield of oil obtained in the filtrate was 81.2% and the yield of stearin removed as press cake was 18.8%, based on the decolorized material charged to the filter. The yield in the decolorizing tower was 97% overhead and 3% bottoms or foots. The

original brown grease charged to the decolorizer had a color too dark to read by the F. A. C. system (Fat Analysis Committee). The overhead from the decolorizer had a color of 2.9 red in a 5 inch Lovibond column. The 3% foots obtained were of the consistency and color of ordinary pitch. This pitch contained 59½% moisture and volatile matter, 13½% total fatty acids, 1.3% nitrogen and 0.32% phosphorus, the remainder being insoluble matter. The original brown grease had an iodine number (Wijz) of 68.0. The stearin cake from the filter had an iodine number of 27.7, and the filtrate from the filter had an iodine number of 78.0 and a pour point of 40° F. The titer of the stearin was 50.1° C.

As another example of the operation of the invention, hydrolyzed white grease fatty acids which were too dark to read on the F. A. C. color system were subjected to a propane decolorizing operation in a column using 30 volume propane to 1 of oil. The temperature was about 174° F. with a 3° F. temperature gradient and the pressure was about 475 pounds per square inch. The yield of decolorized fatty acids with a color not darker than 1 F. A. C. was 97.3%. The bottoms or foots fraction was black and viscous and contained about 10% total fatty acids. The decolorized overhead fraction from the tower was subjected to a depropanizing operation whereby the propane to oil ratio was reduced to about 4 to 1 at a temperature of about 120° F. and at a pressure of about 265 pounds. Heat was supplied in the depropanizing operation by an electrical heating coil. The material was then chilled by gradually reducing the pressure to about 45 pounds per square inch. The temperature was reduced to about 12° F. with an accompanying reduction of the propane ratio to about 3.6 to 1 of oil. The material was filtered and the products stripped of propane. The resulting red oil fraction had an iodine number of 99½, a titer of about 3° C. and a color not darker than 3 F. A. C. The stearin or stearic acid removed from the filter had an iodine number of 3.4 and a melting point of 124° F. The yield of red oil was 60.6% and of stearic acid was 39.4% on the basis of the overhead decolorized product.

Soybean fatty acids obtained by the acidulation and hydrolyzing of soybean refining foots may be subjected to a decolorizing operation as above described and the product subjected to a subsequent chilling and filtration at a propane ratio of 5 to 1 and temperature of about -20° F. to -30° F. An overhead fraction with an iodine number of about 90 and a liquid filtrate fraction with an iodine number of around 165 are obtained.

Sperm oil may be decolorized in a manner similar to that described above and the propane to oil ratio reduced from 20 to 1 to about 3.5 to 1 in the depropanizing operation. A slow release of an additional ½ volume of propane will chill the solution from 120° F. to a temperature of 10° F. suitable for filtering. A yield of about 92.9% of sperm oil with a maximum pour point of 40° F. and 7.1% of spermaceti with an iodine number of 15.2 and a melting point of 108° F. are obtained.

Other materials which may be treated substantially as described above include edible tallow, lard, cottonseed oil, coconut oil, soybean oil, sardine oil, whale oil of the glyceride variety, dark inedible fallows, yellow grease, white grease, hydrogenated fish oils, and the methyl esters and fatty acids of such products. The latter materials

as well as those mentioned in the above examples are included within the meaning of the term "fatty material" which as used in the claims is intended to mean fats, fatty oils, and fatty acids, or mixtures thereof, and methyl esters of said fats and fatty oils. The term "fat" as used in the claims is intended to mean solid fats or fatty oils of animal, vegetable, or marine origin.

While we have described the invention in connection with the use of propane as solvent other normally gaseous hydrocarbons such as ethane, propylene, iso and normal butanes and mixtures thereof may be used.

The present invention has the advantage of decolorizing and fractionating products in one continuous operation without the necessity of changing solvents. Also the energy required to compress the normal gaseous hydrocarbon into the liquid state for the decolorizing operation is more advantageously utilized in this process by the subsequent refrigeration operation in the fractionation step. For example, on account of the relatively high temperature conditions in the decolorizing operation, a large amount of energy is required to maintain the propane in the liquid phase. This excess energy is advantageously utilized for chilling the decolorized product and to crystallize the higher melting point fractions in the subsequent fractionation operations. Moreover, in the process of the present invention the solvent is more efficiently used and may be recovered with a minimum amount of equipment and power facilities.

Obviously, many modifications and variations of the invention hereinbefore set forth may be made without departing from the spirit and scope thereof, and, therefore, only such limitations should be imposed as are indicated in the appended claims.

We claim:

1. A process for treating fatty materials which comprises subjecting a solution of the fatty material in a normally gaseous hydrocarbon solvent to elevated temperature and pressure conditions whereby two liquid phases are formed, the lighter of said phases containing relatively light color fatty material and the heavier of said phases containing relatively dark color materials, separating the phases, vaporizing at least a portion of the normally gaseous hydrocarbon in the lighter phase by reducing the pressure while controlling the heat content of the remainder of the solution so that the temperature thereof does not fall below about 100° to 135° F., chilling the solution of light color fatty material sufficient to precipitate a higher melting point fraction and separating said higher melting point fraction from the lower melting point fractions.

2. A process for refining and fractionating fatty materials which comprises forming a solution of the fatty material in a normally gaseous hydrocarbon solvent, raising the temperature of the solution to around 170° to 185° F. to form a lighter refined liquid phase and a heavier liquid phase containing impurities while maintaining pressures sufficiently high to retain the normally gaseous hydrocarbon in the liquid phase, separating the phases, reducing the pressure in the lighter phase sufficiently to vaporize at least a portion of the normally gaseous hydrocarbon thereby cooling the unvaporized portion to around 100° to 135° F., chilling said unvaporized portion to around -20° to +40° F. to crystallize a higher melting point fraction, and separating the crystals from the refined product.

3. A process for refining and fractionating fats, which comprises mixing with the fat about 20 to 30 volumes of a liquefied normally gaseous hydrocarbon, subjecting the resulting solution to temperatures sufficiently high to cause the separation of a phase containing impurities while under pressures sufficiently high to maintain said hydrocarbon substantially liquefied, separating said phase, vaporizing a portion of the liquefied normally gaseous hydrocarbon from the remaining solution until about 4 to 5 volumes of hydrocarbon remain while controlling the heat in said solution so that it is not chilled below about 100° F., continuing said chilling sufficient to cause the crystallization of a fraction of the fat and separating said crystallized fraction from the remainder of the fat.

4. A process for refining and destearinizing fats which comprises subjecting the fatty material in the presence of liquefied propane to temperatures near the critical point of the propane whereby two liquid phases are formed, one of said phases containing decolorized fatty material and the other phase containing foots, separating the foots, reducing the pressure in the decolorized phase to around 200 to 300 pounds thereby vaporizing the propane and cooling the solution while controlling the temperature whereby the temperature does not fall below about 100° F., chilling said solution by further reducing the pressure to about 30 to 75 pounds to cause the stearin to crystallize and filtering the solution to separate the stearin.

5. A process for refining and fractionating fatty acids which comprises forming a solution of the fatty acids in about 15 to 30 volumes propane, subjecting said solution while under pressure sufficiently high to maintain the propane in the liquid phase to temperatures of about 170° to 185° F. at which the product separates into two phases, one of said phases containing refined

fatty acids and the other phase containing impurities, separating the phases, chilling the refined fatty acid phase by utilizing the heat of vaporization of the propane to temperatures sufficiently low to crystallize high melting point fatty acids, said chilling taking place in two stages, the temperature in the first stage being reduced to about 100° to 135° F. and in the second stage to about -20° to +40° F., and filtering said crystallized fatty acids from the solution.

6. A process for refining and fractionating fatty materials which comprises subjecting a solution of the fatty material in a normally gaseous hydrocarbon solvent to temperature and pressure conditions to cause a separation of a heavier liquid phase containing impurities and a lighter liquid phase containing decolorized fatty material, separating said phases, reducing the pressure to vaporize a portion of said hydrocarbon from the lighter liquid phase while supplying heat to avoid cooling below about 100° F., chilling the decolorized fatty material by vaporizing a further quantity of the normally gaseous hydrocarbon to a temperature sufficiently low to crystallize the higher melting constituents of said fatty material and then filtering the mixture to separate said higher melting point constituents.

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