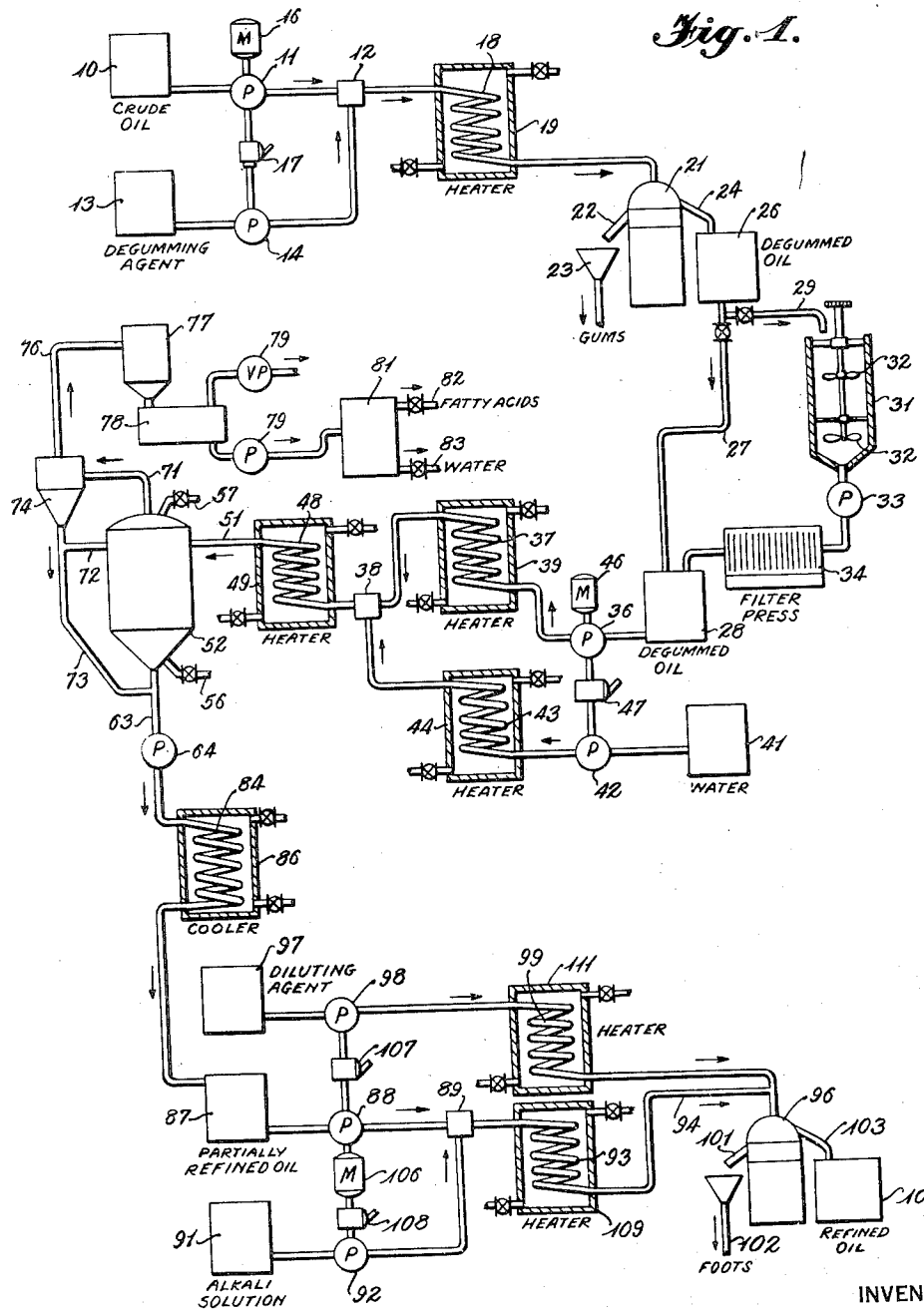
Aug. 2, 1949. B. CLAYTON 2,478,089

PROCESS OF REFINING GLYCERIDE OILS

Filed Oct. 10, 1945 2 Sheets-Sheet 1

Fig. 1.



INVENTOR

Benjamin Clayton

BY Harris, Kiech, Foster & Harris

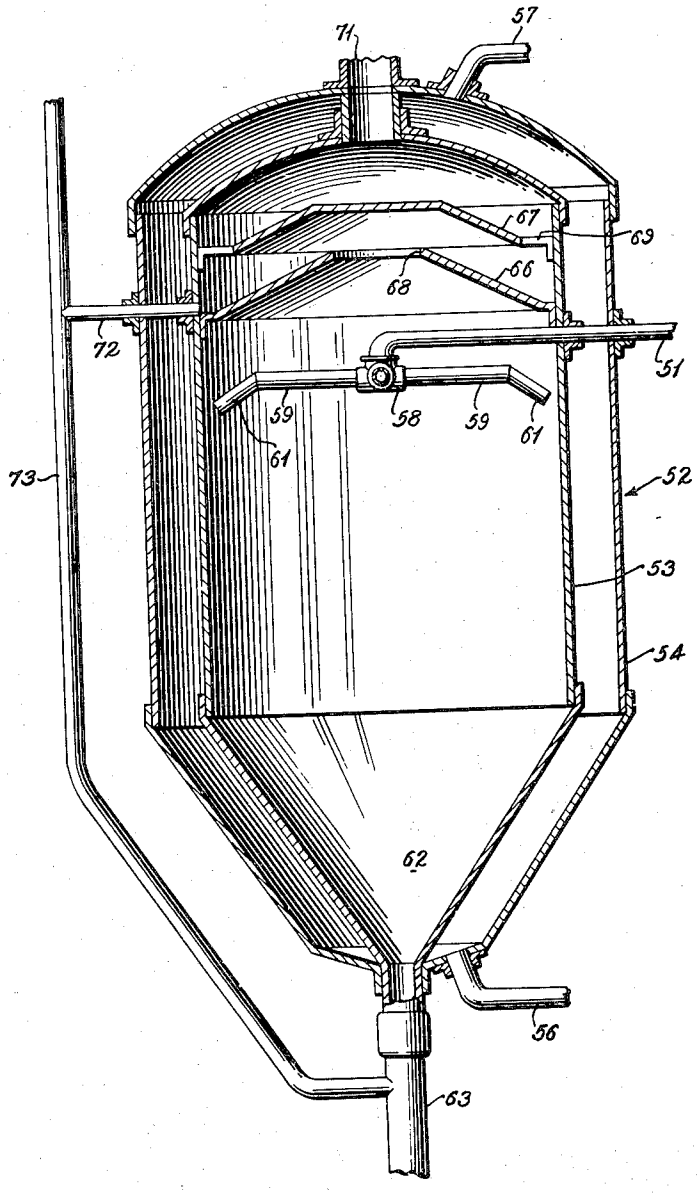
ATTORNEYS

Aug. 2, 1949. B. CLAYTON 2,478,089

PROCESS OF REFINING GLYCERIDE OILS

Filed Oct. 10, 1945 2 Sheets-Sheet 2

Fig. 2.



INVENTOR
Benjamin Clayton
BY Harris, Kech Foster & Harris
ATTORNEYS

Patented Aug. 2, 1949 2,478,089

UNITED STATES PATENT OFFICE

2,478,089

PROCESS OF REFINING GLYCERIDE OILS

Benjamin Clayton, Houston, Tex., assignor, by mesne assignments, to Benjamin Clayton, Houston, Tex., doing business as Refining, Unincorporated

Application October 10, 1945, Serial No. 621,410

6 Claims. (Cl. 260—428)

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This invention relates to a process of refining glyceride oils and more particularly to an improved process in which free fatty acids are removed from the oil by separating the fatty acid in vapor form from the oil.

The most commonly employed refining process for glyceride oils involves the treatment of the crude oil to neutralize the free fatty acids in the oil with an aqueous alkali solution to form soap and subsequent separation of the resulting soapstock from the oil. Present-day continuous processes have reached a high stage of efficiency such that they produce excellent refined oils with low losses. Nevertheless, the fatty acids, which constitute a valuable by-product, are recovered in saponified form in conjunction with various other impurities in the oil. To recover high quality free fatty acids, it has been necessary to acidulate the soap and then further purify the fatty acids by various processes including distillation.

The distilling of the free fatty acids from crude vegetable oils in the presence of steam has also been suggested. Such processes have, however, had certain disadvantages which rendered them less desirable than the alkali refining processes referred to. For example, distillation of the free fatty acids has required that the oil be maintained at high temperatures for extended periods of time in contact with water or wet steam. Thermal decomposition and polymerization of the oil as well as of impurities in the oil occurred to lower the quality of the oil and considerable splitting of the glycerides to produce less valuable fatty acids also took place. The latter factor increased the losses of neutral oil and also extensive losses were produced by entrainment of neutral oil in the vapors removed therefrom. Furthermore, fatty acid distillation processes do not sufficiently remove the free fatty acids and leave other undesirable residual impurities. This has required further treatment of the oil to remove such residual impurities and the operations which have been available for this purpose have resulted in excessive losses of neutral oil.

In accordance with the present invention, high quality fatty acids are separated in vapor form from the oil in a rapid continuous process and are then condensed in the form of high quality relatively pure fatty acids. In order to accomplish this, the crude glyceride oils as first carefully treated to remove substantially all of the mucilaginous materials commonly known as gums, these gums containing, among other things, phosphatides, proteins, etc. With certain highly colored oils, for example, cottonseed

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oil, the crude oils from which the gums have been removed are also subjected to a decolorizing treatment prior to removal of free fatty acids, if a light colored end product is required. This is necessary as the high temperatures employed in the high temperature fatty acid vaporizing steps tend to set the color in the oil rendering it difficult, if not impossible, to later remove this color.

The glyceride oil treated as above described is then subjected to a fatty acid vaporizing step. This step preferably includes the continuous mixing of a stream of water with a stream of the oil in a closed system under conditions of elevated temperatures and pressures and then a rapid lowering of the pressure on the stream of oil and water so that steam is generated in situ throughout the oil to assist in vaporizing free fatty acids. The vaporized fatty acids and steam are then separated from the oil under vacuum conditions and condensed. The condensed fatty acids are easily separated from the condensed aqueous material by difference in specific gravity to recover high quality fatty acids. The entire fatty acid distillation step is carried out in a rapid and continuous manner so that the oil is subjected to high temperatures for extremely short periods of time, thus avoiding thermal degradation of the oil.

The oil from which fatty acids have been removed as above described is then treated to remove residual impurities by an alkali treatment step. In many cases, it is desired to reduce the color of the oil and in any event, the oil from the fatty acid distillation steps usually contains deleterious quantities of unsaponifiables and other impurities, for example, sterols, sterol glucosides and analogous materials, which are desirably removed. Also, any residual free fatty acids in the oil may be substantially completely removed. The residual impurities, including color bodies, unsaponifiables and free fatty acids, may be effectively removed by treating the oil with caustic alkali solutions and subjecting the resulting mixture to centrifugal separation.

Oils which are low in free fatty acid and gum content are extremely difficult to treat adequately with alkalies as small amounts of soap produced in the alkali treatment tend to separate with the oil instead of the aqueous phase in a centrifugal separation. That is to say, caustic alkali solutions in sufficient amount and concentrations to adequately remove the desired impurities tend to cause stratification of the material in the centrifugal into three phases instead of two, namely, an oil phase, an intermediate phase constituting

a mixture of oil, soap and water, and a heavier aqueous phase containing most of the impurities. Any adjustment of the centrifugal which produces a clean oil effluent causes substantial losses of neutral oil as part of the heavier effluent from the centrifugal and similarly, any adjustment of the centrifugal which reduces the oil losses causes a substantial amount of soap to be delivered in admixture with the lighter or oil effluent. Attempts to use dilute solutions have not been successful as the impurities are not adequately removed and neutral oil losses become excessive. Excellent separation of impurities with low losses can be obtained by employing relatively concentrated alkali solutions and delivering substantial amounts of water into the centrifugal separator so as to mix with the aqueous phase therein. A clean oil effluent can thus be obtained without substantial losses of neutral oil in the aqueous effluent. Even diluting the mixture just prior to centrifugal separation provides an improved separation although the losses of neutral oil due to emulsification into the aqueous phase are somewhat greater than is the case when the diluting water is admixed with the heavier material only in the centrifugal.

It is therefore an object of the invention to provide an improved process of refining glyceride oils in which the fatty acids are continuously separated from the oil in vapor form.

Another object of the invention is to provide a complete process for refining glyceride oils in which various types of impurities originally present in the oil are separately recovered and in which high quality fatty acids are a direct product of the process.

Another object of the invention is to provide an improved process of refining glyceride oils in which free fatty acids are separated from the oil in vapor form in a rapid continuous process and refining losses are minimized.

A further object of the invention is to provide a process of refining glyceride oils in which free fatty acids are vaporized from the oil under high temperature conditions and in which the process is rapidly and continuously carried out to avoid thermal degradation of the oil.

A still further object of the invention is to provide a process of refining glyceride oils in which water is thoroughly admixed with the oil and steam rapidly produced throughout the oil to assist in vaporizing the free fatty acids from the oil.

Other objects and advantages of the invention will appear in the following description of a preferred embodiment thereof, suitable apparatus for which is shown in the attached drawings, in which

Figure 1 is a schematic diagram of apparatus for carrying out the entire refining process; and

Figure 2 is a somewhat diagrammatic view in vertical section of the vapor separating chamber of Figure 1.

Referring more particularly to the drawings, crude glyceride oil may be continuously pumped from a source of supply shown as a tank 10 by means of a proportioning pump 11 to a mixer 12. A stream of a degumming agent, ordinarily water or a dilute solution of an electrolyte, may be simultaneously pumped from a tank 13 by means of a proportioning pump 14 to the mixer 12. Any known or suitable proportioning mechanism may be employed, that illustrated in Figure 1 being shown merely for purposes of illustration. The proportioning mechanism illustrated may include

positive displacement pumps 11 and 14 driven by a variable speed electric motor 16, a variable speed device 17 being illustrated between the motor 16 and pump 14 to vary the proportions of oil and degumming agent. The mixer 12 may be any suitable or known device for producing an intimate mixture in stream flow, a preferred device being that shown in the patent to Thurman, No. 2,142,062, granted December 27, 1938. The pumps 11 and 14 force the resulting mixture from the mixer 12 through any suitable flow heating device, for example, the heating coil 18 positioned in the casing 19 through which any suitable heating medium can be circulated. The mixture is heated in the coil 18 to a temperature facilitating centrifugal separation and is delivered from the coil 18 into a continuous centrifugal separator 21.

The separator 21 may be of any known or suitable type and is preferably a heated centrifugal similar to that shown in the patent to Clayton, No. 2,100,277, granted November 23, 1937. The degumming agent causes the gums to become insoluble in the oil thus enabling separation in the centrifugal. The gums are discharged from the centrifugal as a heavier aqueous effluent through a spout 22 into a receiver 23. An oil substantially free of gums is discharged as the lighter effluent through a spout 24 into a receiver 26.

If the oil being refined does not required decolorizing before vaporization of free fatty acids, it may be directly delivered through a pipe 27 to a tank 28 forming a source of supply for the free fatty acid separation step. Substantially all glyceride oils including corn oil, sesame oil, sunflower oil, soya bean oil, etc., may be treated directly after degumming for removal of free fatty acids. In fact, about the only oil requiring intermediate treatment for color removal to produce a light colored final product is cottonseed oil. Cottonseed oil is an extremely dark colored oil and unless treated for color removal prior to high temperature treatment in the free fatty acid removal step, the color is set in the oil so as to resist removal therefrom. Such a dark colored oil may be delivered from the tank 26 through the pipe 29 into a decolorizing chamber 31. In the decolorizing chamber 31 the oil may be agitated by means of agitator 32 with a small amount of a color adsorbent, for example, acid treated clay, fuller's earth, etc., and then delivered by means of a pump 33 through a filter press 34 into the tank 28.

The oil collected in the tank 28 either directly from the tank 26 or from the filter press 34 may be withdrawn by means of a proportioning pump 36 and forced through a heating coil 37 to a mixer 38, the heating coil 37 being positioned in a chamber 39 through which any desired heating medium may be circulated. Water may be withdrawn from the tank 41 by means of a proportioning pump 42 and forced through a heating coil 43 to the mixer 38, the coil 43 also being positioned in a casing 44 through which any desired heating medium may be passed. The pumps 36 and 42 may form part of a proportioning mechanism including a variable speed motor 46 and a speed change device 47. The pumps 36 and 42 should be capable of developing relatively high pressure so that the water and oil can be mixed in the mixer 38 under such high pressures.

The mixer 38 may be of the same type as the mixer 12 previously described. An intimate mixture of oil and water is produced in the mixer 38 and this mixture is then forced by the pumps 36 and 42 through a heating coil 48 positioned in

the casing 49 through which any desired heating medium may be passed. As discussed in more detail below, the oil and water are preferably mixed in the mixer 38 under a pressure sufficiently high to prevent substantial evaporation of water at the mixing temperature. A rapid decrease in pressure occurs near the discharge end of the heating coil 48 so that the water as well as free fatty acids are to at least a substantial extent vaporized in this coil prior to delivery of the resulting mixture through a pipe 51 into a vapor separating chamber 52.

The vapor separating chamber 52 is shown in more detail in Figure 2 and may include an inner chamber 53 surrounded by a casing 54 forming a heating jacket for the chamber 53. Any desired heating medium may be passed through the heating jacket by means of pipes 56 and 57. The pipe 51 preferably terminates within the inner chamber 53 in a distributor 58 having a plurality of radially extending distributor pipes 59 terminating in downwardly inclined nozzles 61 which direct the heated mixture from the pipe 51 downwardly and against the heated walls of the chamber 53. This liquid constituent of the mixture, namely, the glyceride oil, is thus directed against the walls of the chamber 53. The liquid content will ordinarily be in the form of fine droplets suspended in the vapors at the time of contact with the walls of the chamber and these droplets coalesce to form films of liquid flowing down the heated walls of the chamber 53. Vaporized free fatty acids and water are largely separated from the oil droplets by this action but additional free fatty acids can vaporize from the thin films of liquid on the heated walls of the chamber 53 in the presence of steam with which the chamber 53 is largely filled. The oil substantially free of water and free fatty acids collects in the lower portion 62 of the chamber 53 and is withdrawn through a pipe 63 by means of a pump 64 (Figure 1).

Some glyceride oil may be entrained as small droplets in the steam and fatty acid vapors in the chamber 53 and such droplets of oil are largely removed from the vapors by means of baffles 66 and 67. That is to say, the vapors flow rapidly upwardly through a central aperture 68 in the baffle 66 and make an abrupt change of direction so as to flow outwardly and downwardly between the baffles 66 and 67. These vapors then make another abrupt change of direction so as to flow upwardly through apertures 69 spaced around the periphery of the baffle 67. The vapors then flow inwardly and upwardly above the baffle 67 and out of the vapor separating chamber through a pipe 71. Entrained particles are thrown out of the vapors by the abrupt changes of direction and collect upon the outer periphery of the baffle 68. The collected liquid may be discharged from the vapor separating chamber 52 through a pipe 72 connecting with a pipe 73 also connected with the pipe 63 for the treated oil.

As shown in Figure 1, the vapors discharged from the vapor separating chamber through the pipe 71 may be delivered to an entrainment separator 74 for further separation of entrained liquid. The entrainment separator 74 may be of any known or suitable type such as the well-known cyclone separator, liquid separated from the vapors in the separator 74 flowing downwardly through the pipe 73 into the pipe 63. Water and fatty acid vapors substantially free of entrained liquid may then be delivered through the pipe 76 to a condenser 77 communicating with a receiver 78. A vacuum pump 79 may be connected to the receiver to maintain a vacuum in the receiver 78, condenser 77, entrainment separator 74 and vapor separating chamber 52. Condensed water and fatty acids may be removed from the receiver 78 by a pump 79 and delivered to a settling tank 81 in which the water and fatty acids separate by difference in specific gravity. Fatty acids may be withdrawn through a pipe 82 and water through a pipe 83. Separation of fatty acids and water by settling can be efficiently carried on in the present process as emulsifying agents are substantially absent although emulsion breaking agents may be employed, if found necessary, and any other suitable separation operation such as centrifugal separation may be employed, if desired.

The oil removed from the vapor separating chamber 52 will ordinarily contain a small amount of residual free fatty acids. It may have a darker color than that desired and will ordinarily contain substantial amounts of other impurities not vaporized in the vapor separating chamber. Such impurities may include sterols, sterol glucosides, etc., usually considered to be undesirable in refined oils. These impurities, including color bodies and residual free fatty acids, can be largely removed from the oil by treating the same with a caustic alkali solution. The oil pumped from the vapor separating chamber 52 is ordinarily at a relatively high temperature and it is desirable to rapidly cool the oil both to prevent thermal degradation thereof and to condition the oil for the alkali treatment. This oil is therefore preferably immediately passed through a cooling coil 84 positioned in a casing 86 through which any suitable cooling medium may be passed and after cooling the oil may be delivered to a tank 87. The cooled oil may be withdrawn from the tank 87 by means of a pump 88 and delivered to a mixer 89 which may be of the same type as the mixer 12. A caustic alkali solution may be simultaneously withdrawn from a tank 91 by means of a proportioning pump 92 and also delivered to the mixer 89. The resulting mixture may then be passed through a heating coil 93 and delivered by a pipe 94 into a continuous centrifugal separator 96.

The concentration and amount of alkali solution added to the oil is usually sufficient to cause stratification in the centrifugal separator 96. A substantial amount of diluting agent, which is ordinarily water, may be added to the mixture in the pipe 94 just prior to entrance of the mixture into the centrifugal 96 although a preferred operation is to deliver the diluting agent into the interior of the centrifugal bowl so as to mix substantially only with the aqueous layer therein. Thus, a centrifugal separator of the type disclosed in my copending application, Serial No. 483,953, filed April 21, 1943, now Patent 2,412,251 is the preferred type of separator employed. In any case, the diluting agent may be withdrawn from a tank 97 by means of a proportioning pump 98 and is preferably delivered through a heating coil 99 to the centrifugal separator 96. In the centrifugal separator, the mixture is separated and a heavier aqueous phase containing the impurities is discharged through a spout 101 into a receiver 102, the refined oil being discharged as the lighter effluent through a spout 103 into a tank 104. The proportioning mechanism including the pumps 88, 92 and 98 may be similar to that described above and may include a variable speed motor 106 directly connected to

the oil pump 88, speed change devices 107 and 108 being positioned between the motor and the pumps 88 and 92, respectively. Also, the heating coils 93 and 99 may be positioned in casings 109 and 111, respectively, through which any desired heating medium may be passed.

The degumming operation of the present invention may be carried out substantially in accordance with the patent to Thurman No. 2,150,732, granted March 14, 1939. That is to say, water or a very dilute solution of substantially any electrolyte which will not react with the oil or free fatty acids may be admixed in the mixer 12, and the mixture heated in the heating coil 18 and centrifugally separated in the separator 21. The amount of degumming agent will ordinarily range between 1 and 10% of the oil depending upon the amount of gums present in the particular oil and mixing will ordinarily be carried on at atmospheric temperatures. This mixture is then heated to an appropriate separation temperature for the particular oil in the coil 18, the separation temperatures ordinarily being between approximately 100 and 160° F.

If decolorization is required, substantial decolorizing may ordinarily be accomplished by admixing .5 to 1% of adsorbent, preferably acid treated clay, with the oil in the tank 31 and then passing the oil through the filter press 34. It is of course understood that a plurality of tanks 31 and filter presses 34 may be provided so that they may be employed alternatively to enable the other portions of the process to be carried on continuously. It is also understood that a plurality of centrifugals may be employed in both the degumming and the final refining operations instead of the single centrifugals 21 and 96, respectively, in order to increase the capacity of the refining system. The temperature of the oil in the decolorizing step will ordinarily be that of the oil discharged from the centrifugal 21, namely, a temperature between 100 and 160° F.

Oil withdrawn from the tank 28 by means of the pump 36 will ordinarily be heated in the heating coil 37 to a temperature between 200 and 300° C., the preferred temperature ranging between 225 and 275° C. prior to being delivered to the mixer 38. Similarly, water withdrawn from the tank 41 by means of the pump 42 will ordinarily be heated in the heating coil 43 to approximately the same temperature as the oil is heated in the coil 37. It is preferred to maintain the pressure in the mixer 38 above that at which substantial amounts of water will evaporate at the temperature specified. This means that pressure in the mixer 38 will range from approximately 220 pounds per square inch for 200° C. up to approximately 1235 pounds per square inch for 300° C. For the preferred temperatures ranging between 250° C. and 275° C., these pressures will fall between approximately 570 and 855 pounds per square inch in order to prevent evaporation of water in the mixture. These pressures are the pressures of saturated steam at the respective temperatures, i. e., the temperatures are the boiling points of water at the respective pressures. That is to say, the pressure at which water will boil at the temperature employed.

Since the discharge portion of the heating coil 48 is connected to the interior chamber 53 of the vapor separating chamber 52 and this interior is operated under vacuum conditions, the pressure will drop progressively through the coil 48. This pressure drop will not be uniform as relatively large volumes of steam and fatty acid vapors are formed in the coil so that the velocity of the material flowing through the coil increases as vapor is formed therein. The pressure drop will therefore be largely concentrated in the discharge portion of the coil so that it is relatively easy to maintain high pressures in the mixer 38. In fact, the coil 48 functions as a pressure balance such that the pressure in the mixer 38 changes rather slowly as the speed of the pumps 36 and 42, i. e., the throughput through the mixer and the coil 48, is varied. The proportion of water and oil delivered to the mixer 38 will vary in accordance with the amount of free fatty acid contained in the oil. Thus, the amount of water will ordinarily not be less than the amount of free fatty acids in the oil and may be three or four times the amount of free fatty acids. For oils ranging from .5% free fatty acids up to 10%, the amount of water will range from approximately 1% to 20%.

By preheating both the oil and the water to a relatively high temperature prior to mixing, i. e., temperatures as high as 275 to 300° F., a large amount of heat is imparted to these liquids which is available for vaporizing water and fatty acids. This is true since the temperature of the resulting mixture of vapors and liquid in the vapor separating chamber will ordinarily range between approximately 150 and 200° C. Additional heat may also be imparted to the mixture in the coil 48 to furnish heat for vaporizing water and fatty acids. It is possible to perform all of the heating in a single long coil or series of coils 48 after forming the mixture of oil and water. This, however, results in a longer time of contact between the oil and water at elevated temperatures and is, therefore, less desirable.

The mixer 38 intimately admixes the water with the oil in the form of extremely fine droplets so that in the coil 48 steam is formed and brought into contact with substantially all portions of the oil. The fatty acids vaporize readily into the steam at the low pressures in the discharge end of the coil, the rapid movement of the mixture through the coil maintaining the steam intimately admixed with the oil. As the volume of steam and fatty acid vapors becomes large, the mixture changes to a fog of glyceride oil droplets in the vapors either in the coil or immediately upon discharge into the inner chamber 53 of the vapor separating chamber and the oil droplets are impinged at high velocity against the heated walls of the chamber. The vapors are thus separated from the majority of the droplets and the droplets coalesce to form a film of liquid flowing down the interior chamber 53 so that fatty acid or water remaining in the liquid readily escapes as vapor into the interior of the chamber. The walls of the interior chamber 53 are preferably maintained substantially at or slightly above the average temperature of the liquid and vapors in the chamber by means of the heating jacket 54, i. e., at a temperature between approximately 150 and 200° C., so as to furnish heat to the liquid on the walls for further vaporization.

As described above, substantially all entrainment is removed from the vapors by the baffles 66 and 67 in the vaporizing chamber and by the entrainment separator 74 before these vapors are delivered to the condenser 77. The condenser 77 is ordinarily employed to condense both the fatty acids and the water although a plurality of condensers may be employed, if desired, for fractional condensation so as to at least partially separate the free fatty acids from the water. The

condenser 77 in conjunction with the vacuum pump 78 is employed to produce a relatively low vacuum in the interior chamber 53 of the vapor separator, for example, an absolute pressure ranging from ¼ to 1 inch of mercury. The best results are obtained by the highest vacuum which it is practical to maintain, for example, pressures below ¼ inch of mercury absolute.

The entire fatty acid removal step is preferably carried on as rapidly as possible and the treated oil rapidly cooled in the coil 84 in order to maintain the time at which the oil is at the high temperature as short as possible. All of the heating and cooling coils are preferably made of relatively small diameter pipe with as thin wall sections as possible for the pressure maintained therein. Thus, the entire heating, vapor separating and cooling operations on any given portion of oil will ordinarily not exceed five minutes and is preferably of shorter duration. It is particularly desirable to maintain liquid water in contact with the oil at high temperatures for as short a time as possible to avoid any action of the water upon the oil to form additional free fatty acids i. e., this time should be sufficiently short to prevent substantial hydrolysis of the oil. Ordinarily the contact of the oil and liquid water at high temperatures can be made as short as one minute or less. By the fatty acid removal step, the free fatty acid content of the oil can be reduced to an extremely small amount, for example, .1% or less in a rapid continuous process with substantially theoretical losses and without degrading the oil.

As treatment with alkaline solutions for removal of residual impurities is usually most effective at low temperatures, the oil will ordinarily be cooled in the coil 84 to a temperature below 100° F., for example, temperatures between 70 and 90° F. The amount of alkali added to the oil in the mixer 89 will depend upon the nature and amount of residual impurities in the oil including the free fatty acids. The best results are usually obtained with highly concentrated caustic alkali, for example, solutions of caustic soda ranging between 20° and 50° Bé. although in some instances more dilute solutions down to approximately 12 to 14° Bé. may be employed. Glyceride oils vary so greatly in properties depending upon the type of oil, the climatic conditions under which the oils were produced and the method of obtaining the crude oil from the oil-bearing seeds that no precise concentration or amount of alkali solution can be given. This is particularly true when it is realized that the present process is also applicable to animal oils including fish oil. Depending upon the oil being treated, the amount of caustic solution will ordinarily range between 1 and 5%, the concentrations ordinarily ranging between approximately 20 and 50° Bé.

Separation of the aqueous phase from the oil is usually more effective at higher temperatures than the mixing temperatures and the mixture may therefore be heated in the coil 93 to a temperature ranging between 100 and 160° F. depending upon the particular oil being treated. If stratification difficulties occur in the centrifugal 96, sufficient water may be delivered into the mixture or into the aqueous layer in the centrifugal as above described. This water is preferably heated in the coil 99 to at least the temperature of the mixture entering the centrifugal and it is many times advantageous to heat the water to a higher temperature, for example, temperatures close to 212° F., particularly when the water is added to the aqueous layer only in the centrifugal.

Stratification difficulties are caused by the low or negligible gum content of the oil being treated and by the high concentration of alkali solution frequently required for adequate action upon impurities from the oil, the high concentrations being particularly advantageous when color bodies are being removed from the oil. Since there are substantially no gums present to retain the soap emulsified in the aqueous layer, it is salted out by the high concentration of the aqueous layer and appears in the separated oil unless the aqueous layer in the centrifugal is diluted until the soap remains therein. Since the different oils to be treated vary greatly as discussed above, no precise ranges of concentrations and amounts of water to be used on any particular oil can be specified. Ordinarily the skilled refiner in practicing the present invention increases the concentration of the alkali solution until the oil is substantially free of impurities originally present in the oil and at the same time increasing the amount of water added until the oil is substantially free of soap. As a guide for determining the dilution required best results are usually obtained when the concentration of alkali in the heavier effluent is below 10 to 12° Bé. and in some cases concentrations as low as 6 to 8° Bé. may be necessary. By employing relatively highly concentrated solutions of caustic alkali such as caustic soda and then diluting in the centrifugal, removal of residual impurities including color bodies can be accomplished with extremely low losses.

In carrying out the invention as above described, the gums are first removed, as the presence of gums renders the fatty acid distillation process inefficient and also certain constituents of the gums tend to decompose or degrade at the high temperatures employed, thus decreasing the quality of the oil. The gums are, however, a valuable by-product and are recovered separately from the fatty acids and from other impurities. The fatty acids are directly recovered from the oil in substantially pure form and constitute another valuable product. Furthermore, the aqueous phase separated from the oil by the centrifugal 96 of the alkali treatment step contains unsaponifiables including certain oil-soluble vitamins as well as materials such as sterol glucosides. These materials are valuable by-products and occur in relatively high concentration in the foots from the centrigual 96. They are relatively easily recovered from the foots, for example, the foots may be partly acidulated with a mineral acid such as sulfuric or hydrochloric, the amount of acid employed being just sufficient to neutralize the free alkali without substantial decomposition of soap. Under these conditions, the valuable constitutents collect in a discrete aqueous layer which can be separated by decantation. The small amount of residual soap may then be further acidulated to recover a rather good grade of fatty acids, if desired.

While I have disclosed the preferred embodiment of my invention, it is understood that the terms thereof may be varied within the scope of the following claims.

I claim:

1. The process of removing free fatty acids from a glyceride oil containing said free fatty acids, which process comprises, forming at a temperature between 200° and 300° C. a confined

flowing stream of an intimate mixture of said glyceride oil and an amount of water between approximately 1 and 4 times the amount of said free fatty acids in said glyceride oil, discharging said stream into a vapor-separating chamber having a vacuum therein at a time not more than approximately 5 minutes after the forming of said mixture at said temperature as a mixture of liquid glyceride oil and water and fatty acid vapors, the time during which said water is in contact with said oil at said temperature being sufficiently short to prevent substantial hydrolysis of said oil, withdrawing said vapors from said chamber at a rate sufficient to maintain an absolute pressure therein at least as low as one-quarter inch of mercury, condensing said vapors and recovering fatty acids, and separately withdrawing liquid glyceride oil from said chamber and promptly cooling said glyceride oil.

2. The process of removing free fatty acids from a glyceride oil containing said free fatty acids, which process comprises, forming at a temperature between 200° and 300° C. a confined flowing stream of an intimate mixture of said glyceride oil and an amount of water between approximately 1 and 4 times the amount of said free fatty acids in said glyceride oil, maintaining said confined stream under a pressure above that at which water boils at said temperature, discharging said stream into a vapor-separating chamber having a vacuum therein at a time not more than approximately 5 minutes after the forming of said mixture at said temperature as a mixture of liquid glyceride oil and water and fatty acid vapors, the time during which said water is in contact with said oil at said temperature being sufficiently short to prevent substantial hydrolysis of said oil, withdrawing said vapors from said chamber at a rate sufficient to maintain an absolute pressure therein at least as low as one-quarter inch of mercury, condensing said vapors and recovering fatty acids, and separately withdrawing liquid glyceride oil from said chamber and promptly cooling said glyceride oil.

3. The process of removing free fatty acids from a glyceride oil containing said free fatty acids, which process comprises, forming at a temperature between 200° and 300° C. a confined flowing stream of an intimate mixture of said glyceride oil and an amount of water between approximately 1 and 4 times the amount of said free fatty acids in said glyceride oil, maintaining said confined stream under a pressure above that at which water boils at said temperature, rapidly lowering the pressure on said stream as it advances to form a resulting stream of droplets of glyceride oil suspended in water and fatty acid vapors, discharging said resulting stream into a vapor-separating chamber having a vacuum therein at a time not more than approximately 5 minutes after the forming of said mixture at said temperature, the time during which said water is in contact with said oil at said temperature being sufficiently short to prevent substantial hydroylsis of said oil, withdrawing said vapors from said chamber at a rate sufficient to maintain an absolute pressure therein at least as low as one-quarter inch of mercury, condensing said vapors and recovering fatty acids, and separately withdrawing liquid glyceride oil from said chamber and promptly cooling said glyceride oil.

4. The process of removing free fatty acids from a glyceride oil containing said free fatty acids and which is substantially free from gums, which process comprises, forming at a temperature between 200° and 300° C. a confined flowing stream of an intimate mixture of said glyceride oil and an amount of water between approximately 1 and 4 times the amount of said free fatty acids in said glyceride oil, maintaining said confined stream under a pressure above that at which water boils at said temperature, discharging said stream into a vapor-separating chamber having a vacuum therein at a time not more than approximately 5 minutes after the forming of said mixture at said temperature as a mixture of liquid glyceride oil and water and fatty acid vapors, the time during which said water is in contact with said oil at said temperature being sufficiently short to prevent substantial hydrolysis of said oil, withdrawing said vapors from said chamber at a rate sufficient to maintain an absolute pressure therein at least as low as one-quarter inch of mercury, condensing said vapors and recovering fatty acids, supplying sufficient heat through the walls of said chamber to maintain a temperature therein between approximately 150° to 200° C., and separately withdrawing liquid glyceride oil from said chamber and promptly cooling said gylceride oil.

5. The process of removing free fatty acids from a glyceride oil containing said free fatty acids, which process comprises, forming at a temperature between 200° and 300° C. a confined flowing stream of an intimate mixture of said glyceride oil and an amount of water between approximately 1 and 4 times the amount of said free fatty acids in said glyceride oil, maintaining said confined stream under a pressure above that at which water boils at said temperature, discharging said stream into a vapor-separating chamber having a vacuum therein at a time not more than approximately 5 minutes after the forming of said mixture at said temperature as a mixture of liquid glyceride oil and water and fatty acid vapors, the time during which said water is in contact with said oil at said temperature being sufficiently short to prevent substantial hydrolysis of said oil, the last-named mixture being directed against the inner surface of a wall of said chamber so that said liquid glyceride oil flows as a film down said wall, withdrawing said vapors from said chamber at a rate sufficient to maintain an absolute pressure therein at least as low as one-quarter inch of mercury, condensing said vapors and recovering fatty acids, supplying sufficient heat through said wall to maintain the temperature of said surface between approximately 150° and 200° C., and separately withdrawing liquid glyceride oil from said chamber and promptly cooling said glyceride oil.

6. The process of removing free fatty acids from a glyceride oil containing said free fatty acids, which process comprises, heating a confined stream of water to a temperature between 200° and 300° C. at a pressure above that at which said water boils, heating a confined stream of said glyceride oil to said temperature, intimately mixing said streams at said temperature and under said pressure to form a combined stream containing water in an amount between approximately 1 to 4 times the amount of free fatty acids in said oil, rapidly lowering the pressure on said combined stream as it advances to form a resulting stream of droplets of said oil in water and fatty acid vapors, discharging said resulting stream into a vapor-separating chamber having a vacuum therein at a time not more than approximately 5 minutes after said mixing, the time

during which said water is in contact with said oil at said temperature being sufficiently short to prevent substantial hydrolysis of said oil, withdrawing said vapors from said chamber at a rate sufficient to maintain an absolute pressure therein at least as low as one-quarter inch of mercury, condensing said vapors and recovering fatty acids, and separately withdrawing liquid glyceride oil from said chamber and promptly cooling said glyceride oil.

BENJAMIN CLAYTON.

REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
Re.20,838	Clayton et al.	Aug. 30, 1938
619,020	Henderson	Feb. 7, 1889
1,368,148	Heyerdahl	Feb. 8, 1921
1,385,660	Bodman	July 26, 1921
2,156,863	Mills	May 2, 1939

Dedication

2,478,089.—*Benjamin Clayton*, Houston, Tex. PROCESS OF REFINING GLYCERIDE OILS. Patent dated Aug. 2, 1949. Dedication filed June 30, 1964, by the assignee, *Benjamin Clayton, doing business as Refining, Unincorporated.*

Hereby dedicates to the public the terminal part of the term of said patent effective December 31, 1963.

[*Official Gazette September 29, 1964.*]