This invention relates to a process and apparatus for refining glyceride oils, particularly animal and vegetable oils containing acidic impurities reactive with an alkali to produce separable foams or soapstock. The acidic impurities of such oils include, and are usually composed largely of free fatty acids. These must be removed in the refining operation to produce a marketable oil. Generally stated, the invention is concerned with the refining of such oils by use of soda ash employed in such manner that carbon dioxide is evolved and removed before the oil-soapstock mixture is separated.

An early refining process mixed caustic soda with the oil to react with the acidic impurities and produce soaps which were separable from the oil. Oil losses were relatively large in batch-type operations, and even the later continuous process produced undesirably high losses due largely to the reaction between the caustic soda and the oil itself or to undesirably high entrainment of oil in the separated soapstock.

More recently, soda ash refining processes have been used on many oils. Here aqueous solutions of soda ash have been mixed with the oil in amounts greatly in excess of those theoretically required to react the acidic impurities. Soda ash is a substantially non-saponifying neutralizing agent but when it reacts with fatty acids or with the reaction products of the two, carbon dioxide is liberated and interferes with both the desired separation and the desired completion of the reaction.

To solve this problem, it has heretofore been common to employ the soda ash either in a dehydration-rehydration process or a high-excess process. In the former, an amount of soda ash equal to 5-6 times the amount required to neutralize the oil acidity is employed commercially. The soda ash solution is mixed with the oil and the mixture is sent to a dehydration zone maintained under high vacuum to liberate and remove carbon dioxide and water. The dehydrated mixture is then rehydrated and centrifuged. In the high-excess process the amount of soda ash is sufficient substantially to suppress the formation of carbon dioxide and sufficient to effect a good separation. Excesses of 6-8 times the amount required to neutralize are commonly employed, often more. The oil-soapstock mixture is not dehydrated and rehydrated but is sent directly to a centrifuge. Sufficient excesses of soda ash are used to prevent gassing and to insure that the neutralization of the free fatty acids shall proceed to the bicarbonate state.

In contradistinction to these prior processes, it has been found that substantially improved results can be obtained by employing soda ash in amounts insufficient to prevent liberation of carbon dioxide and then degasifying the mixture before separation. Usually the amount of soda ash will be less than three times the amount theoretically required to neutralize the acidic impurities and preferably not more than about twice the amount thus theoretically required. While such small excesses have been proposed in processes where soapstock separation was effected under substantial pressure and at quite low temperatures in a closed or hermetically sealed centrifuge to avoid liberation of carbon dioxide, such centrifuges are costly and any low-excess-process employing them is sometimes erratic and fails to give the desired products and economies of the present process.

In the prior dehydration-rehydration and high-excess soda ash processes, the resulting soapstocks often present problems. If such soapstocks are to be acidulated, the amount of sulfuric acid required is sometimes greater than is economically justified. The present process permits substantial savings in this regard. In addition, such prior soapstocks are often so low in total fatty acids as not to be saleable, particularly when refining oils low in free fatty acids and high or medium in gum content; also in refining degummed oils low in free fatty acids. Additionally, such soapstocks often have undesirably high moisture content. The present invention produces improved soapstocks of decreased total fatty acid content and moisture content—soapstocks which are excellently suited as additives to animal feeds. It is an object of the present invention to produce a soapstock of low moisture content, typically in the range of 25-40% moisture and preferably less than about 30% moisture.

In my prior application Serial No. 481,930, filed January 14, 1955, I have disclosed a process employing soda ash in low excesses and in amount insufficient to suppress the evolution of carbon dioxide, the carbon dioxide being removed in a chamber in which the pressure was above atmospheric. While that process has many advantages as compared with the older dehydration-rehydration or high-excess processes, it has been found that even better results can be obtained by adjusting the operating conditions and equipment to maintain a slight vacuum in the chamber in which the carbon dioxide is separated. Experience with excesses of 2.5 to 3 times has shown that removal of carbon dioxide under slight pressure is difficult on oils having a free fatty acid content of 1.5% or higher, such a process tending to produce an effluent oil unduly high in impurities, e.g., containing more than 0.2% free fatty acids, and an effluent soapstock unduly high in free oil, e.g., containing upwards of 15-20% free oil. Re-refining of such effluent oils with caustic soda produces unduly high saponification and entrainment losses. When treating crude oils containing 2.0% or more free fatty acids, it is most difficult to obtain effluent oils containing less than 0.2% free fatty acids when separating the carbon dioxide under slight pressure, although the operation becomes quite feasible with the present process. For the most efficient and desirable operations, the effluent oil should contain not more than 0.1% free fatty acids, whether or not it is subsequently re-refined, and the present process makes this commercially possible.

It is an object of the present invention to mix with a glyceride oil an amount of soda ash insufficient to prevent evolution of carbon dioxide, and to separate the carbon dioxide from the resulting oil-soapstock mixture under a slight vacuum. Another object of the invention is to maximize the removal of carbon dioxide while minimizing the removal of water from the oil-soapstock mixture. In the preferred practice of the invention, the amount of water left in the mixture is sufficient to produce a centrifugally separable soapstock. If a part of the water is removed with the carbon dioxide, the soapstock may not remain to solubilize and make separable the soapstock, thus resulting in soapstock entrainment in the separated oil. Also, removal of a part of the water with the carbon dioxide would tend to create a foam in the degasser unless all of the water is removed, which is undesirable in the low excess process exemplified.

It has been found possible, in accordance with the
The present invention, to obtain the advantages of low-excess operation while avoiding the need for rehydration. This has been made possible by separation of carbon dioxide under such vacuum conditions as will remove all of the carbon dioxide almost without any significant dehydration.

The carbon dioxide that is liberated when using soda ash in low excess must be rather completely removed if it is not to interfere with separation in the centrifuge. If the carbon dioxide is removed from the mixture while under slight pressure, there is a tendency for some of the carbon dioxide to remain dissolved in the oil and to be liberated at any lower pressure existing in the centrifuge. By use of the present process and apparatus the removal of the carbon dioxide is more complete, leading to better separation and to other improved results.

Unless all of the carbon dioxide is removed, the reaction between the soda ash and the acidic impurities will not go to completion. This is particularly important in processes employing soda ash in low excess. It is difficult to remove all of the carbon dioxide and force the reaction to completion if the carbon dioxide is separated under superatmospheric pressure. However, by separating under a slight vacuum the carbon dioxide can be substantially completely removed to permit the reaction to go to completion.

The removal of the carbon dioxide in accordance with the present invention destabilizes and deforms the oil-soapstock mixture or emulsion. While it has been previously known that such an emulsion could be destroyed by evaporating the water, the present invention makes it possible to destabilize the mixture or emulsion even while the moisture is present. The resulting mixture will thus separate better and will produce substantially improved products.

If the carbon dioxide is separated under pressure, there is an increased tendency to form acid soaps which are quite soluble in the oil. Such acid soaps have a tendency to form when limited amounts of soda ash are used and result from the combining of one molecule of sodium soap with a fatty acid molecule. If the carbon dioxide is removed completely, as by separating under the slight vacuum as herein contemplated, there is less likelihood of difficulty from such acid soap either because less of it forms or because if formed it tends to be broken by the complete removal of the carbon dioxide.

In accordance with the invention, the effectiveness of removal of the carbon dioxide is also facilitated by spreading the oil-soapstock mixture on a surface as a film. It is an object of the present invention to spread such a mixture on the internal surface of a degasser container to facilitate the complete separation of carbon dioxide therefrom. During flow along such a surface the separation is made more complete because of the thinness of the film, the increased area of the mixture exposed to the gaseous atmosphere, and the increased time of exposure to such atmosphere into which the carbon dioxide may escape.

A further object is to spread the mixture on a heated surface in such manner as to maximize the separation of carbon dioxide while minimizing the removal of moisture.

On the other hand, the degasser of the invention is useful in a wide variety of other processes where it is desired to separate gases, including vapors, from a liquid. In this respect, it is an object of the invention to provide a degasser in which a stream of the incoming liquid is spread uniformly along a surface, preferably the internal surface of a container, this surface being adjustable in temperature by being jacketed. A further object is to apply the liquid to an upright surface in successive sweeps so timed that a substantially uniform film of the liquid flows downwardly along the surface by gravity while exposed to the adjacent atmosphere within the container. A further object is to spread the liquid on an internal wall of a container maintained under a vacuum.

The degasser of the invention provides different processing zones in which different temperatures can be maintained. In addition, it is well adapted to the handling of effluents of a heavy or viscous character and provides an effluent withdrawal means effectively sealing the container and permitting continuous withdrawal of the effluent irrespective of pressure conditions within the container. It is an object of the invention to provide a degasser having one or more such features.

Further objects and advantages of the invention will be evident to those skilled in the art from the following description of the exemplary embodiments, including those suggested in the attached drawing in which:

Fig. 1 is a pipeline diagram of an apparatus for the continuous refining of glyceride oils, illustrating the degasser in vertical section;

Fig. 2 is a horizontal sectional view taken along the line 2—2 of Fig. 1; and

Fig. 3 is a fragmentary view of one of the nozzles of the degasser, taken as indicated by the arrow 3 of Fig. 2.

Referring particularly to Fig. 1, the exemplary apparatus includes, generally described, a proportioning-heating means 10, a degasser 11 and a separating means 12. This apparatus is adapted to refine a glyceride oil withdrawn from a tank 18 by a proportioning pump 19 which delivers a stream thereof through a heater 20 to a mixer 21. Similarly, a solution of soda ash may be withdrawn from a tank 22 by a proportioning pump 23 which forces the stream through a heater 24 and through a pipe 25 which may discharge directly into the mixer 21 or, as shown, to a pipe junction of a pipe 26 which feeds the mixer. In this way, the soda ash solution preliminarily mixes with the oil at such a pipe junction before entering the mixer 21.

The mixer 21 may be of any suitable type which will thoroughly and intimately mix the soda ash solution with the oil. However, when practicing the low excess soda ash process herein disclosed, the soda ash solution should be intensely and rapidly mixed with the oil in the mixing tank. Batch mixing in large kettles is not effective in such process and it is desirable to use mechanical mixing of proportioned streams continuously delivered to a rotary mixer. This mixer 21 may be of this type and the mixing action therein should be so intimate that the agent is thoroughly consumed.

The excess of surface solution is under the control of the proportioning pumps 19 and 23, which can be driven by a motor 27 connected to one of the pumps through a speed-change device 28. Similarly, the motor 27 may drive, through a speed-change device 29 an auxiliary proportioning pump 30 to be later mentioned.

The oil-soapstock mixture issuing from the mixer 21 is advanced under the pressure of the proportioning pumps through a heater 32 and thence to the degasser 11 through a pipe 33. The heaters 20, 24 and 26, as well as similarly shown heaters to be later mentioned, may be of the type including a pipe coil 34 disposed in a housing 35 through which any defined heating medium is circulated by means of pipe connections 36.

The time of mixing or the time of contact before reaching the degasser 11 is not critical. It is usually desirable, however, that this time of contact be at least about three minutes, measured from the initial mixing to the time of discharge into the degasser. This time of contact may be provided by the mixer 21 and the heater 32. Should it be desired to further increase this time of contact or additionally to adjust the temperature of the oil-soapstock mixture before delivery to the degasser 11, a valve 38 in the pipe 33 may be closed to divert the flow through a now-open valve 39 in a pipe 40 leading to a
mixture 41 which may discharge through a heater 42 before returning the fluid to the pipe 33 beyond the valve 38. If it is desired to introduce the soda ash solution in two portions, as is sometimes beneficial, a first portion is introduced through the pump 23 and a second portion may be withdrawn from the tank 22 through a valve 45, being proportioned by the auxiliary pump 30. The resulting stream may be delivered through a heater 46 to a pipe 47 which joins the pipe 48 ahead of the mixer 41. In this way the second portion of the soda ash solution can be mixed with the previously formed oil-soapstock mixture advancing in the pipe 46, the mixer 41 serving to mix the second portion of the solution, the temperature of the mixture being adjusted by the heater 42 if this is desired.

The degasser 11 includes a generally upright container 50 supported by a suitable framework 51. In the preferred construction, the container 50 is divided into several jacketed sections arranged for rapid temperature adjustment of the interior liquid if this is desired. As shown, the container 50 includes an upper section 53, a tapered section 54 and a lower section 55.

The upper section 53 is cylindrical, being closed by a head 57. It provides an inner circular surface 58 surrounded by a jacket 59 which may be filled with any suitable fluid through pipes 60. Additional pipes 61 circulate a fluid medium through a coil 62 within the jacket. This medium is usually a heating medium.

The incoming stream, exemplified as an oil-soapstock stream containing carbon dioxide, is conducted to the interior of the container 50 through a pipe 64 connected to a head 65 which supplies the liquid to the interior of a pipe 66 through holes 67 therein. The pipe 66 is rotatably mounted in the head 65 and in a bearing 68 outside the container, being driven by bevel gears 69 from a gear-reduction or variable speed motor 70. The lower end of the pipe 66 carries a T 71 from which arms 72 extend, these arms being shown as having curved ends providing nozzles 73. This structure represents a rotary distributor which distributes the incoming liquid as a film on the heated circular surface 58. The nozzles 73 move in recurring paths immediately adjacent the inner circular surface 58 and provide orifices which discharge the liquid at a velocity approximately the same as the lineal or peripheral velocity of the moving nozzles. Discharge at a slightly higher velocity is preferred. The moving nozzles deposit the liquid on the surface 58 in contact with a previously-supplied portion of the liquid, applied to the surface on a previous fractional rotation of the distributor but which have moved by gravity along the surface since being initially supplied thereto. By controlling the rate of rotation in this manner, a continuous film of the liquid is formed, this film gravitating along its supporting surface 58 and being exposed to the atmosphere at the center of the container 50. In the exemplified process, this atmosphere is predominantly carbon dioxide which has separated from such film.

The construction of the nozzles 73 to obtain the desired film-forming action is not critical. As shown in Figs. 2 and 3, these nozzles are formed by flattening and sidewardly cutting the ends of the pipes 72 to provide horizontally elongated openings 74 facing and moving close to the circular surface 58. If desired, a spreader plate 75 may be secured to the pipe ends, providing an arcuate surface 76 facilitating the desired spreading action. In other instances, spray type nozzles may be employed instead of the spreader-type nozzles illustrated.

It is desirable in the exemplified process that the carbon dioxide be separated from the oil-soapstock mixture under a slight vacuum. This slight vacuum can be produced by forcefully withdrawing the carbon dioxide from the upper section 53 of the container 50, using a suitable pump, blower or fan 80 connected to the container by a pipe 81. A dry-type vacuum pump can be employed and it is sometimes feasible to employ merely a fan or blower to exhaust the carbon dioxide at a sufficient rate to maintain the desired slight vacuum. One or more sight glasses 82 may be provided in the head 57, permitting internal inspection of the degasser during operation.

The film flowing down the heated circular wall 58 reaches a conical wall 85 of the tapered section 54, flowing therealong until it joins a body 86 of the liquid. During such flow along the conical wall, the film may be rapidly heated or cooled or it may be merely maintained at the existing temperature. This is made possible by use of a correspondingly-tapered jacket 87 providing a space which can be filled with any suitable liquid through pipes 88, the space containing a coil 89 through which a heating or cooling medium may be circulated by use of the pipes 90 shown.

The lower section 55 of the degasser 11 comprises a cylindrical housing 92 surrounded by a jacket 93 equipped with a coil 94 to heat or cool the surrounding liquid, introduced through pipes 95, thus heating or cooling the liquid or mass moving downwardly within the housing 92. Such downward movement is induced by turning a conveyor screw 96 driving snugly within the cylindrical housing 92 and journalled by bearings 97 and 98 to turn about the central axis of this housing. The bearing 98 may be mounted in a suitable cross-member 100 of the framework 51 at a position below the housing 92.

The lowermost end of this housing is closed by a plate 101 carrying a packing gland 102 which seals the central shaft of the conveyor screw 96. This central shaft is preferably a pipe 105 driven by a motor 106 through suitable gearing 107.

To increase the heat-transfer action of the lower section 55, a heating or cooling medium may be circulated internally of the screw conveyor 96. Such a medium may be supplied under pressure to a pipe 110 which discharges to a constricted portion 111 traversing a chamber 112 formed by a manifold 113. The constricted portion 111 is joined with a pipe 115 smaller in external diameter than the internal diameter of the pipe 105. The uppermost end of the smaller pipe 115 opens on the interior of the larger pipe 105 near the bearing 97. Liquid forced through the pipe 110 thus rises in the smaller pipe 115 and returns in the annular space between the two pipes, collecting in the chamber 112 from whence it is removed through a pipe 116.

The internal and external heating or cooling of the liquid or mass to be withdrawn produces a very flexible operation, particularly if used in conjunction with the variable speed motor 106. The viscosity of the liquid can be rapidly adjusted by change in temperature, the viscosity and the speed of rotation of the conveyor screw 96 determining the pumping action exerted on the liquid or mass being drawn downwardly in the housing 92 and discharged from the lower end thereof through a pipe 117. Indeed, it is possible continuously to withdraw liquids from the degasser against even a high-vacuum therein while still maintaining the interior of the container sealed from the atmosphere.

In the exemplified low-excess soda ash process, the mixture of oil and soapstock, free of carbon dioxide but substantially unreduced in water content, flows through the pipe 117 under the control of a valve 118 therein. The temperature may be further adjusted by flow through a heat exchange device 119 before entering the centrifugal means, exemplified as a centrifuge 120 representative of a bank thereof usually employed in commercial processes. In the exemplified process, each centrifuge may be of the conventional type operating at atmospheric pressure. In certain circumstances, however, it may be of the closed or pressure type effecting centrifugal operation under super-atmospheric pressure. In either event, it separates the mixture into streams of oil and soapstock respectively discharging through pipes 121 and 122. A rather thick soapstock will often be produced by the
low excess soda ash process herein exemplified. If a thinner soapstock is desired, water or aqueous solutions can be added just ahead of or within the centrifuge 120 as by being supplied through a pipe 124 to the zone of separation to discharge with the soapstock. Such aqueous solutions may also act as weightings or soapstock stiffening agents but are ordinarily not needed in the preferred practice of the invention.

Exemplifying further the low excess soda ash process of the invention, I prefer to use excesses of 1.5-2 times the amount theoretically required to react with the acidic impurities of the oil. In some instances, excesses up to 3 times can be employed in practicing such process. Use of the lowest possible excesses of soda ash is sometimes important from the standpoint of decreased costs. For example, some 360 lbs. of soda ash can be saved in refining a tank car of oil containing 2.1% free fatty acids when employing an excess of 1.5 times, as compared with a 3 times excess. However, the use of the lowest feasible excess is desirable for other reasons such as reducing the volume of the soapstock; lowering the refining loss of oil through occlusion in the soapstock; production of soapstock of improved quality for certain important uses, including Hass as feed additives; reduced evolution of carbon dioxide which must be withdrawn; etc.

Typically soda ash solutions of 10-26° Bé are employed in the exemplified process, a 20° Bé solution being common. The strength of the solution is determined in part by the desired moisture to be in the soapstock at the time of separation, if no additional moisture is added and if there is no dehydration in the degasser 11.

It is usually desirable that the temperatures in the upper section 53 of the degasser 11 be elevated, e.g., 150-210° F. A large part of this heat is desirably supplied by the heater 20, 24 or 46 before being mixed. The mixture can be further heated in the heaters 32 and 42 or one or both of these heaters can be employed to supply all of the heat needed by the degasser.

The surface 58 on which the mixture is filmed is desirably at a temperature somewhat above that of the oil-soapstock mixture expelled by the rotating nozzles 73. At the same time, if there is to be no significant dehydration of the mixture in the degasser, the temperature of the surface should not exceed the boiling point of water at the slight vacuum existing in the degasser. Often the temperature of the liquid in the jacket 59 may be close to or slightly above such boiling point, being sometimes as high as 250° F. If there is to be significant heating in the degasser, it is beneficial to the complete removal of the carbon dioxide from the mixture that this mixture be spread as a thin film on a heated surface. On the other hand, if the incoming mixture is already at the desired elevated temperature, the jacket 59 may be used to keep the surface 58 at the same temperature to prevent chilling in the upper section 53 of the degasser 11.

The wall temperature in the tapered or intermediate section 54 of the degasser may be equal to, higher or lower than in the upper section 53. Equality of such temperatures is often used in the low-excess soda ash process although it is possible to use the higher wall temperatures in the section 54 if there is to be progressive heating in the degasser.

In the lower section 55 of the degasser the defoamed and decarbonated mixture is kept mixed by the screw 96. The temperature here depends in part upon the desired viscosity of the mixture to be discharged as an effluent, also upon the degree of heating or cooling desired at this stage before separating the soapstock. In some instances, improved operation results from a sudden chilling of the mixture in the lower section 55 even though it may be desirable to further heat the mixture in the heater before the centrifugal separation, which will usually be in a range of 160-220° F., typically 180-210° F. If centrifugal separation in the usual atmospheric-pressure centrifuges is effected. However, if desired, such separation can take place in centrifuges of the closed or pressure type discharging streams of oil and soapstock under superatmospheric pressure, the pressure increase from the degasser to the centrifuges being produced by the screw conveyor effluent means of the degasser or by an auxiliary pump in the pipe 117.

In practicing the low-excess soda ash process, a slight vacuum is maintained in the upper interior or central zone of the degasser so that the carbon dioxide separates from the film of the mixture at subatmospheric pressure. In the absence of substantial throttling of the mixture in the nozzles 73 this slight vacuum may extend back into the pipe 64. The slight vacuum in the degasser 11 is usually about 0.5-10" of mercury, preferably 1-4" of mercury.

When processing certain oils of high gum content, it is sometimes necessary to increase the amount of soda ash above the amount that would be used on an otherwise corresponding oil of low gum content. With high-gum oils some of the soda ash appears to be absorbed by the gums so as not to be available for reaction with the free fatty acids, and this should be taken into account before selecting the low excesses on the bases herein disclosed and claimed.

The degasser 11 of the invention finds utility in processes other than the low-excess soda ash process herein exemplified. Thus, for example, it can be used with the medium or high-excess soda ash processes either as a substitute for presently used dehydrators in the dehydration-rehydration process or to remove carbon dioxide which may result from improper mixing of higher excesses of soda ash with a crude oil, particularly with oils of high free fatty acid content. When used as a substitute for the present dehydrators in the dehydration-rehydration process, the degasser has the outstanding advantage of reducing the likelihood of priming because of sudden changes in vacuum. For example, one of the primary sources of such priming is a sudden change in vacuum from, say, 28.5" to 15" of mercury as a result of cooling water failure or reduced flow in the steam ejector system normally employed to maintain the vacuum. Upon restoration of the high vacuum, part or all of the contents of the conventional dehydrator may be sucked out of the chamber. When using the present degasser at such high vacuum, such priming is eliminated. This effect is aided in part by the large internal zone of the degasser and the distance between the body 86 and the pipe 81 leading to the vacuum pump.

The degasser 11 may also be used for quickly and effectively drying degummed oils or oils that have been water washed following degumming or alkali refining. Some crude cottonseed oils are sensitive to color setting and treatment thereof can be improved by removing the water under vacuum during flow as a film along cooled internal surfaces of the degasser, e.g., the inner surface 58 or the conical wall 85. A dry, quick-cooled degummed cottonseed oil produced in this fashion is stable and when subsequently refined with caustic soda will produce a refined oil of light color which will not revert.

When the degasser is used in high-vacuum processes, the vertical disposition of the sections and the versatility and rapidity with which temperature changes are effected will be found to make possible improved operation altogether. Also, the internally jacketed screw conveyor 96 makes possible a rapid heat transfer with the material moving downward within the degasser. This screw conveyor can be employed to withdraw a viscous effluent continuously even under high-vacuum conditions within the degasser.

Various changes and modifications can be made with-
out departing from the spirit of the invention as defined in the appended claims.

I claim as my invention:

1. A process for refining glyceride oils containing acidic impurities including free fatty acids, which process includes the steps of: mixing soda ash with said oil in the presence of water under conditions producing an oil-soapstock mixture containing carbon dioxide and water, the amount of soda ash being insufficient to prevent formation of carbon dioxide in the mixture; removing the carbon dioxide from the mixture in the absence of substantial dehydration of the mixture while the mixture is under vacuum; and then subjecting the resulting mixture to a separating action to separate the soapstock from the oil.

2. A process for refining glyceride oils containing acidic impurities including free fatty acids, which process includes the steps of: mixing soda ash with said oil in the presence of water under conditions producing an oil-soapstock mixture containing carbon dioxide and water, the amount of soda ash being insufficient to prevent formation of carbon dioxide in the mixture; removing the carbon dioxide from the mixture in the absence of substantial dehydration of the mixture while the mixture is under vacuum; and then subjecting the resulting mixture to a separating action to separate the soapstock from the oil.

3. A process for refining glyceride oils containing acidic impurities including free fatty acids, which process includes the steps of: mixing soda ash with said oil in the presence of water under conditions producing an oil-soapstock mixture containing carbon dioxide and water, the amount of soda ash being insufficient to prevent formation of carbon dioxide in the mixture; removing the carbon dioxide from the mixture in the absence of substantial dehydration of the mixture while the mixture is under vacuum; and then subjecting the resulting mixture to a separating action to separate the soapstock from the oil.

4. A process for refining glyceride oils containing acidic impurities including free fatty acids, which process includes the steps of: mixing oil with an amount of soda ash less than three times the amount thereof theoretically required to neutralize said acids, said amount of soda ash no more than three times the amount thereof theoretically required to neutralize said acids, said amount being insufficient to suppress the evolution of carbon dioxide, thus forming an oil-soapstock mixture containing carbon dioxide and water; moving a stream of said mixture into a closed chamber and forming same into a film flowing along a surface exposed to the interior of said chamber to facilitate separation of the carbon dioxide from the mixture; withdrawing substantially all of the carbon dioxide from said chamber in substantially moisture-free condition; withdrawing a mixture of oil and soapstock containing water from said chamber; and centrifuging the soapstock of the withdrawn mixture from the oil thereof.

5. A process as defined in claim 6 including the steps of regulating the withdrawal of carbon dioxide and the withdrawal of the oil-soapstock mixture from said chamber to develop a slight vacuum therein.

6. A process as defined in claim 6 in which said chamber has an upright circular surface and an adjacent nozzle, and wherein said stream is formed into a film flowing along said surface by moving said nozzle in a recurring path inside and adjacent said upright circular surface at a given linear velocity while discharging the stream of mixture therefrom at a velocity nearly equal to said given linear velocity to coat the circular surface with a film of the mixture, the carbon dioxide separating from the film of the mixture.

7. A process as defined in claim 8 in which the mixture gravitates downward along said upright circular surface as a film, said nozzle moving about the central axis of said circular surface while moving in said recurring path, the rate of rotation being sufficient to deposit the mixture into contact with a portion of the mixture applied to said circular surface on a previous sweep of the nozzle to form a continuous film of the mixture gravitating along such surface.

8. A process for refining those crude glyceride oils containing at least 1.8% free fatty acids, which process includes the steps of: mixing soda ash with said oil in the presence of water, the amount of soda ash being about 2.5-3 times that required theoretically to react said fatty acids, thus forming a mixture containing soap and carbon dioxide; removing the carbon dioxide from the mixture in the absence of any substantial amount of dehydration by subjecting the mixture to a vacuum while at a temperature below the boiling point of water; and centrifuging the resulting mixture while carbon dioxide is no more than the amount thereof theoretically required to neutralize said acids, said amount

References Cited in the file of this patent

UNITED STATES PATENTS

<table>
<thead>
<tr>
<th>Patent Number</th>
<th>Date</th>
<th>Inventor(s)</th>
<th>Title</th>
</tr>
</thead>
<tbody>
<tr>
<td>2,017,525</td>
<td>May 24, 1936</td>
<td>Stenback</td>
<td>Process for Producing Carbon Dioxide from Oil and Water</td>
</tr>
<tr>
<td>1,297,333</td>
<td>Mar. 18, 1919</td>
<td>Earl</td>
<td>Method of Separating Oil and Water from Oil Soapstock</td>
</tr>
<tr>
<td>2,614,656</td>
<td>Oct. 21, 1952</td>
<td>Clark et al.</td>
<td>Process for Separating Oil and Water from Soapstock</td>
</tr>
<tr>
<td>2,620,894</td>
<td>Dec. 9, 1952</td>
<td>Peterson et al.</td>
<td>Process for Separating Oil and Water from Soapstock</td>
</tr>
<tr>
<td>2,686,796</td>
<td>Aug. 17, 1954</td>
<td>Markley et al.</td>
<td>Process for Separating Oil and Water from Soapstock</td>
</tr>
<tr>
<td>2,710,664</td>
<td>June 14, 1955</td>
<td>Blackmore et al.</td>
<td>Process for Separating Oil and Water from Soapstock</td>
</tr>
<tr>
<td>2,748,884</td>
<td>Aug. 21, 1956</td>
<td>Erwin</td>
<td>Process for Separating Oil and Water from Soapstock</td>
</tr>
<tr>
<td>2,759,957</td>
<td>June 6, 1956</td>
<td>Thurman</td>
<td>Process for Separating Oil and Water from Soapstock</td>
</tr>
<tr>
<td>2,769,827</td>
<td>Nov. 6, 1956</td>
<td>Clayton</td>
<td>Process for Separating Oil and Water from Soapstock</td>
</tr>
</tbody>
</table>
Dedication


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

*[Official Gazette September 29, 1964.]*