Feb. 13, 1940. B. CLAYTON ET AL 2,190,595

REFINING PROCESS

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Fig. 1.

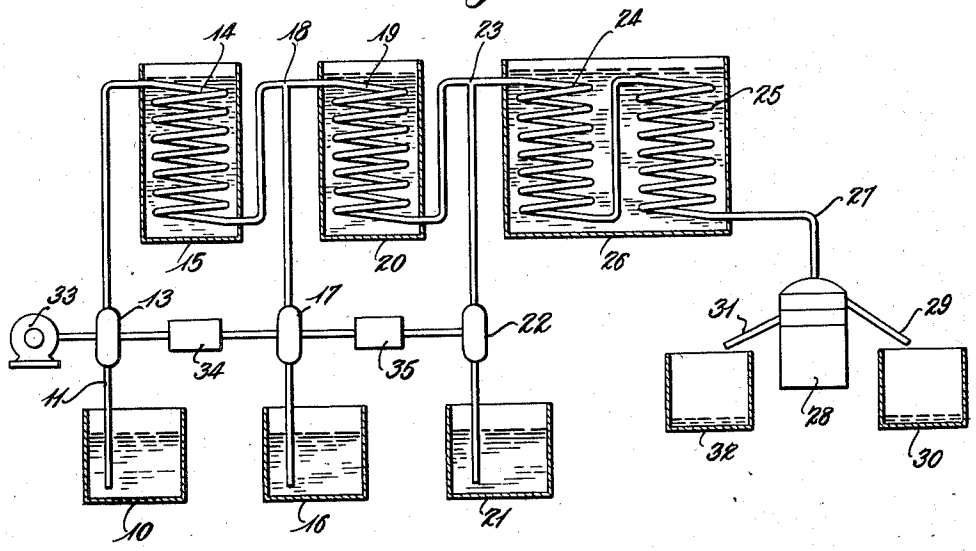
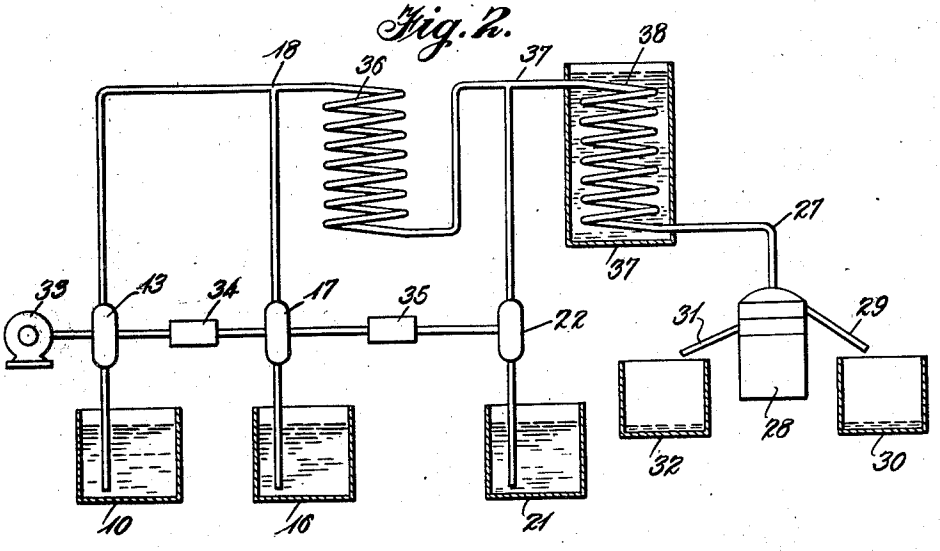


Fig. 2.



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REFINING PROCESS

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Application December 19, 1939, Serial No. 310,082

18 Claims. (Cl. 260—425)

This invention relates to refining processes and more particularly to processes for refining glyceride oils in which centrifugal separation is employed for separating neutral oil from the soap stock resulting from reactions between a refining agent and the free fatty acids and other impurities contained in unrefined glyceride oils.

In such processes, the efficiency of the centrifugal separation step is of primary importance since a substantial quantity of neutral oil is normally entrained with the soap stock and separated therewith. The entrained neutral oil is difficult to recover from the soap stock and represents a serious loss in prior processes. Also an excess of refining agent over that necessary to neutralize the free fatty acids is ordinarily employed in such refining processes. The excess is effective to reduce the color of the oil being refined and is usually maintained in contact with the oil for a period of time greater than that necessary for complete neutralization of the free fatty acids to permit the excess to act upon the colored impurities and also to properly condition the soap stock for separation. The excess tends to also saponify neutral oil during this period, resulting in a further refining loss.

It is an object of the present invention to provide a process of refining glyceride oils by which centrifugal separation is improved and the amount of neutral oil entrained in the soap stock is substantially reduced.

Another object of the invention is to provide a process by which the saponification of neutral oil is also substantially reduced.

Another object of the invention is to provide a process of refining glyceride oils in which a material, which retards the saponification of neutral oils, is incorporated with the mixture of neutral oil and soap stock shortly after the refining agent has been mixed with the unrefined oil.

A further object of the invention is to provide a process of refining glyceride oils in which a heated liquid material, which has no deleterious effect upon the neutral oils, is incorporated with the mixture of neutral oil and soap stock after neutralization but prior to centrifugal separation in order to retard the saponification of neutral oil by the excess of refining agent and to promote the separation of the soap stock from the oil.

A still further object is to provide a process in which a liquid material having a higher specific gravity than the neutral oil is mixed with the neutral oil and soap stock prior to centrifugal separation in order to promote separation of the soap stock from the oil and to retard the adherence of constituents of the soap stock to the walls of the centrifugal bowl.

Other objects and advantages of the invention will appear in the following description of preferred embodiments of the invention which will be described with reference to the apparatus shown in the attached drawing of which:

Figure 1 is a diagrammatic view of an apparatus suitable for carrying out the present invention; and

Figure 2 is a similar view of an apparatus suitable for carrying out a modification of the invention.

The apparatus of Figure 1 is suitable for a continuous refining process employing preheated oil and includes a supply tank 10 for the oil to be refined. Oil is withdrawn from this tank through a pipe 11 and forced by a proportioning pump, indicated diagrammatically at 13, through a preheating coil 14 positioned in a heating bath, for example hot water, contained in the tank 15. A properly proportioned solution of refining agent is withdrawn from a suitable supply tank 16 by the proportioning pump 17 and forced at right angles into the flowing stream of preheated oil at 18. By this operation substantially complete mixing and neutralization of the free fatty acids is accomplished and the mixture of oil and soap stock is forced by the pumps 13 and 17 through a second coil 19 positioned in a heating bath in the tank 20.

A predetermined amount of separation promoting liquid is withdrawn from supply tank 21 by the proportioning pump 22 and forced at right angles into the flowing stream of oil and soap stock mixture at 23 so as to be intimately mixed therewith. The resultant mixture is forced through coils 24 and 25 positioned in a heating bath in the tank 26 and then delivered through the pipe 27 to a centrifugal separator 28. The neutral oil is therein continuously separated from the other constituents of the mixture and forms the lighter effluent which is discharged from the centrifugal 28 by the spout 29 into a receiving tank 30. The soap stock along with the separation promoting liquid forms the heavier effluent and is discharged from the spout 31 into a receiving tank 32. The proportioning pumps 13, 17 and 22 are shown as being driven by an electric motor 33, which may be a variable speed motor, and variable speed devices indicated diagrammatically at 34 and 35 may be employed between the various pumps to adjust the relative proportions of the various materials. If desired the oil heating coil 14 can be omitted and the oil preheated in

the tank 10 or any other type of heating apparatus such as, for example, a chamber provided with an agitator and steam coils or surrounded by a heating jacket can be employed in lieu of coil 14. The temperature to which the oil may be heated before mixing with the refining agent will vary with different oils from approximately 120° to 160° F. but in most cases a temperature of approximately 130° F. has been found desirable.

The refining agent employed is ordinarily an alkali which combines with the free fatty acids to form soap, a solution of caustic soda being the usual refining agent. This solution may be of the concentration known to the prior art as being suitable for the particular oil being treated. The usual excess of refining agent over that theoretically necessary to combine with the free fatty acids is ordinarily employed and this excess acts upon the colored impurities in the oil during the passage of the mixture through the coils 19, 24 and 25 to reduce the color of the refined oil.

The present invention is particularly effective when a de-emulsifying and saponification inhibiting agent is employed in the process since such agents tend to decrease the specific gravity of the heavy effluent. With certain oils it is advantageous to add small amounts of such a de-emulsifying and saponification inhibiting agent. This agent may be incorporated with the refining agent in the tank 16 or otherwise added to the refining agent or the oil to be refined, for example, by injecting the same into a flowing stream of one of these materials before the refining agent is mixed with the oil so that the agent is present during the neutralization of the free fatty acids. One example of such an agent is iso-propyl alcohol, which may be employed in an amount between approximately .25% and 1.75% of the weight of the oil being refined and preferably between 1% and 1.5%. Other de-emulsifying and inhibiting agents are ethyl alcohol, ethyl acetate, iso-propyl acetate and other polyhydric and monohydric alcohols such as butyl, propyl or amyl alcohols and their esters. However, in no event do we employ alcohol in sufficient quantities to substantially completely dissolve the soap component of the soap stock formed in the process as we are cognizant of the fact that such complete dissolving of the soap component of the soap stock would render the mixture incapable of satisfactory centrifugal separation due to the stratification of the soap stock components so as to cause separation of part of the soap stock with the oil. These materials are either added in concentrated form or if diluted, the concentration of the original refining solution is adjusted to give a resultant refining solution of the proper concentration for the particular oil being refined. The de-emulsifying and inhibiting agents are effective to retard the saponification of neutral oil and to prevent the formation of tight and difficultly separable emulsions in the refining process.

The present invention is also applicable in processes employing agents having both a neutralizing and de-emulsifying and saponification inhibiting functions such as sodium alcoholate or potassium alcoholate which may be substituted in whole or in part for the usual caustic alkali refining agents.

The refining agent may also be heated prior to admixture with the oil but in most cases this has been found to be unnecessary since the amount of refining agent is relatively small as compared to the amount of oil so that the temperature of the mixture is not materially lower than that of the preheated oil. Also, any heat necessary to maintain the required temperature can be supplied in coil 19 which may be surrounded by water at a temperature somewhat higher than the temperature of the preheated oil. For example, this water temperature may be 140° F. for oils preheated to 130° F. Jetting the refining solution into a flowing stream of the oil and then flowing the mixture through the coil 19 is a particularly effective method of producing an intimate mixture with consequent neutralization of the free fatty acids followed by a reaction period in which color impurities are acted upon by the excess refining agent. Other apparatus may, of course, be employed such as, for example, one or more mechanical mixers or agitators preferably having heating coils or jackets. Any apparatus employed should quickly disperse the refining agent in the oil and maintain the mixture substantially uniform to prevent settling of the soap stock. It should also preferably have sufficient capacity to provide the reaction period above mentioned prior to the addition of the separation promoting agent.

The separation promoting agent may be any liquid having a specific gravity greater than that of the refined oil and which has no deleterious effect upon the oil or soap stock. Water or water solutions of salts which do not react chemically with the oil or soap stock are effective. Alkaline metal salts such as sodium sulphate, acetate, carbonate or silicate or the equivalent potassium salts may be employed. Of these the carbonates or silicates are in many cases desirable since they produce alkaline solutions. When salt solutions are used, their concentration will depend upon the type of oil being treated and the specific gravity found desirable with the type of soap stock produced. This concentration, therefore, contemplates the employment of salt solutions containing a small amount of soluble salts up to substantially concentrated solutions. In no event, however, is the character or quantity of the salt solution sufficient to cause graining out of the soap stock which would interfere with satisfactory centrifugal separation. The water or salt solution may be added to the mixture of oil and soap stock without being heated, but in general better results are accomplished by heating the water or solution prior to addition to a temperature at least as high as that of the mixture to which they are added and preferably to the boiling temperature of water. By adding such a separation promoting liquid, the soap stock is diluted and the heavy porous pasty type of soap stock which results from refining of many types of oils, particularly when caustic alkalis are used, is prevented from forming. The soap stock is thereby prevented from sticking to the bowl of the centrifugal and building up a layer which shifts the neutral zone in the centrifugal resulting in imperfect separation.

Also the occluding of neutral oil in the soap stock being discharged from the centrifugal is to a large extent prevented. It has been found that the net saving in fat going into the soap stock may be as high or even greater than 10% of the weight of the soap stock on a dry basis. That is, the soap stock will normally contain free oil to the extent of 10 to 15% while, if the water or salt solution is added to the mixture

before centrifuging the free oil will be reduced, in most cases, to less than 5%. Thus the yield of refined oil will be increased several per cent. The amount of separation promoting liquid which is added will vary with the nature of the oil being refined and will ordinarily fall within a range of 1% to 10% and usually within the range of 3% to 6%.

The separation promoting solution may be added to the mixture immediately preceding the centrifugal separation step and advantageous results secured but, as shown in Figure 1, it is preferred to mix the solution with the oil and soap stock mixture somewhat before the mixture reaches the entrance of the centrifugal. For example, the separation promoting liquid may be added at the point 23 and the resultant mixture forced through the coils 24 and 25. This is advantageous, particularly where a hot separation promoting liquid is employed as the hot liquid prevents emulsification and dilutes the excess refining agent to such an extent that there is no longer any possibility of a saponifying action on the neutral oil. The separation promoting liquid thereby acts as a saponification inhibiting and de-emulsifying agent as well as preventing the soap stock from forming a tight or semi-pasty and porous condition. At the same time, it aids in causing a better separation of the soap stock from the oil where the tendency of the crude is not to give a clean and definite break-away of the soap stock from the refined oil.

The coils 24 and 25 have as their primary function the conditioning of the soap stock for separation. During the passage of the mixture through these coils, the fine particles of soap stock formed during neutralization tend to collect or agglomerate into large particles and there is some further action of the refining solution upon the colored impurities and adsorption of the color impurities by the soap stock. The separation promoting liquid aids in the conditioning of the soap stock for separation and prevents occlusion of neutral oils in the particles of soap stock. As shown in Figure 1 these coils are ordinarily positioned in the heating bath so as to maintain the temperature thereof at least as high as the mixture entering the coils and in many instances it has been found desirable to increase the temperature of the mixture up to 140° F. or even as high as 160° F. The mixing of the oil and soap stock with the separation promoting liquid by jetting the latter into a flowing stream of the former and then flowing the result of the mixture through heating coils has been found to be an extremely efficient method of accomplishing the desired operations. Other apparatus may, however, be employed such as one or more mechanical mixers or agitators preferably provided with heating means and having sufficient aggregate capacity to provide for the period of time desirable for conditioning the soap stock for separation.

In the centrifugal separation step, the separation promoting liquid separates with the soap stock since it is of higher specific gravity than the oil. Its employment is particularly desirable when an alcohol or similar saponification inhibiting and de-emulsifying agent is employed during neutralization since the alcohol or equivalent agent is ordinarily of less specific gravity than the soap stock resulting in a reduced difference in specific gravity between the heavier and lighter effluents. The separation promoting liquid compensates for this effect by again increasing the specific gravity of the heavier effluent. In a process where no alcohol or equivalent agent is employed it facilitates separation by increasing the difference in specific gravity between heavier and lighter effluents. Although the separation promoting liquid decreases the tendency of the soap stock to stick to the walls of the centrifugal bowl, as is also the case with the alcohol or equivalent inhibiting and de-emulsifying agent when employed, with certain types of oil which produce soap stock which is extremely difficult to separate from the oil, it may be desirable to employ the heated centrifugal disclosed in the copending application, Serial No. 34,258, filed August 1, 1935, now Patent No. 2,100,277, granted November 23, 1937, to further decrease the tendency of the soap stock to stick to the walls of the centrifugal bowl.

As a specific example of a process carried out in the apparatus of Figure 1, a cottonseed crude oil was preheated to a temperature of 130° F. in the coil 14. A refining solution of caustic soda of the proper concentration and proportion for the particular oil being treated was injected into the stream of preheated oil at 18 and the mixture passed through a coil 19. This coil was immersed in a water bath at 140° F. and was 250 ft. in length. The mixture was passed through the coil at a rate sufficient to give a reaction time of 1 and ½ minutes. When the caustic and oil mixture was discharged from this coil, boiling hot water was injected into it in an amount between 3% and 6% of the weight of the oil and the resulting mixture was then passed through coils 24 and 25. These coils were entirely similar to coil 19 and provided three minutes additional time before the mixture was delivered to the centrifugal. The latter two coils were surrounded by a water bath the temperature of which was maintained at approximately 135 to 140° F. The soap stock separated cleanly from the neutral oil and the amount of neutral oil entrained in the soap stock was reduced from a normal content from 10% to 15% to somewhat less than 5%.

The present invention is also applicable to a process in which a refining agent is mixed with the oil at room temperatures. An apparatus such as shown in Figure 2 is suitable for carrying out such a process. In this apparatus oil at room temperature and a refining agent are withdrawn from tanks 10 and 16 respectively by the proportioning pumps 13 and 17 and the refining agent jetted into the flowing stream of oil at 18. The mixture thus formed is flowed through a coil 36 to provide for a reaction period in which the excess caustic acts upon the colored impurities. This coil may also be at room temperature. The refining solution employed may be any of the refining solutions discussed with reference to the process employing preheated oil.

A separation promoting liquid is withdrawn from the tank 21 by the proportioning pump 22 and injected at 37 into the flowing stream of mixture being discharged from the coil 36. This separation promoting liquid may be either water or a salt solution such as also described with reference to the process employing preheated oil. The separation promoting liquid may also be at room temperature but is preferably heated to approximately the boiling point of water before being admixed with the mixture discharged from the coil 36. This heating may be accomplished in tank 21 or a heating coil provided for this purpose. If the water or saline solution is at an

elevated temperature, it tends to prevent the formation of difficultly separable emulsions. The mixture of oil, soap stock and hot water or saline solution is then passed through a coil 38 positioned in a heating bath in the tank 37 wherein the temperature of the mixture is elevated to preferably 130 to 140° F. although this temperature may be as low as 110° F. for oils producing soap stock which is less difficult to separate. As in the previously discussed process, the separation promoting liquid dilutes the soap stock and prevents it from forming a tight or semi-pasty or porous emulsion thus rendering it more easy to separate from the neutral oil in the centrifugal. Advantageous results can be accomplished by adding the separation promoting solution just prior to separation of the soap stock but if added at the point shown in Figure 2, it also dilutes the excess caustic while the soap stock is being conditioned for separation thus cutting down saponification of neutral oil in the coil 38 particularly when no saponification inhibitor and emulsifying agent has been added to the process prior to mixing the neutral oil and refining agent. In either case, the separation promoting liquid increases the specific gravity of the heavier effluents so as to aid in separation of the soap stock from the oil and this is particularly advantageous when alcohol or equivalent inhibiting or de-emulsifying agents have been employed. As in the case of the process employing preheated oil, the mixture discharged from the coil 38 is delivered by the pipe 27 to a centrifugal 28 which separates the soap stock from the neutral oil. The neutral oil is discharged from a spout 31 into a receiving tank 32 and heavier effluent from a spout 29 to a receiving tank 30. The centrifugal 28 may be of the type ordinarily employed in separating a heavier material from a lighter material and in certain cases it may be desirable to use the heated centrifugal of the copending application above referred to. As in the case of Figure 1, the proportioning pumps 13, 17 and 22 may be driven by a variable speed electric motor 33 with variable speed devices 34 between the proportioning pumps.

The apparatus of Figure 2 has been found to be extremely effective in carrying on the process described in connection therewith. However, other types of apparatus may be employed, for example, mechanical mixers or agitators may be used instead of the mixing and reaction means shown in Figure 2. Such agitators or equivalent mechanisms should have heating means where required and sufficient capacity to give the desirable reaction periods. Whatever type of apparatus is employed, it is preferably of a type which will provide for a closed system such that air does not contact the oil or mixture between the oil supply and separation step.

The present invention is applicable to refining of various types of glyceride oils such as cottonseed, soya bean, peanut, linseed, etc. The conditions of refining such as the temperature, length of reaction periods, and amount of separation promoting liquid will vary with the different types of oil being treated and the point of admixture of the separation promoting liquid with the other materials will also vary in accordance with the type of refining employed and the oil being refined.

In any event, the separation promoting liquid will be mixed with the mixture of oil and soap stock after the neutralization operation and prior to centrifugal separation.

From the foregoing it will be understood that the primary purpose of this invention is to provide a process for reducing saponification losses as well as separation entrainment losses. The soap stock is continuously conditioned for centrifugal separation. When caustic alkali is employed as a neutralizing agent, it is possible, with our invention, to employ a relatively small amount thereof, not substantially more than that theoretically required to neutralize the free fatty acids. Ordinarily a large excess is employed for the reason that it is desired to react with the color impurities and also to produce a soap stock having the gum component thereof in a condition for centrifugal separation. This results, however, in substantial saponification and entrainment losses. However, in accordance with our invention, the soap stock produced by the employment of a small amount of caustic or other reagent not greatly in excess of that required to neutralize, and which is of a character incapable of said centrifugal separation, can continuously, in a quick continuous operation be conditioned for such separation by the subsequent incorporation therewith of a stream of water, desirably admixed with an alkaline reagent, such as soda ash, which changes the character of the soap stock into a separable form. Again, the subsequent addition of a stream of hydrating medium with alkaline material therein contained serves to reduce the color quite substantially during the advancement of the stream to the centrifugal separator. Moreover, by reason of this incorporation of additional alkaline material, in aqueous solution, the resultant mixture not only is rendered relatively non-adhesive and conditioned for centrifugal separation but is susceptible of substantially higher heat treatment, to liquefy the same, than would be the case of the conventional caustic produced foots or soap stock.

When employing an aqueous medium comprising soda ash or other alkaline material, the amount thereof may be such that the total alkali employed in the process is at least several times that necessary to neutralize the free fatty acids even though relatively small amounts of neutralizing agent are initially added.

The invention has utility in the refining of glyceride fats or oils and the term "oil" is employed as inclusive of both oil and fat, irrespective of the viscosity of the material.

By the employment if the term "hydrating medium" in our claims, we intend to define and embrace the hydration of the "foots" or "soap stock" to condition the same for improved centrifugal separability as contra-distinguished from a process, such as exhibited in the Brucke Patent No. 2,035,589, wherein alcohol is used in such quantities during separation as to substantially completely dissolve the soap component of the soap stock; and the term likewise is exclusive of the employment of salt solutions sufficient to cause graining out of the soap stock, in either of which events centrifugal separability of the soap stock or foots from the refined oil is impaired.

This application is a continuation in part of our earlier application Serial No. 121,882, filed January 22, 1937.

While we have disclosed the preferred embodiments of our invention, it is understood that the process herein disclosed may be varied within the scope of the following claims.

We claim:

1. In the process of refining glyceride oils, comprising the steps of mixing an alkaline refining reagent with said oil to neutralize the free fatty acids contained therein and to form soap stock, continuously conditioning such soap stock for centrifugal separation by admixing a stream thereof with a stream of hydrating medium and thereafter subjecting the thus conditioned mixture to the act of continuous centrifugal separation.

2. The process as defined in claim 1 in which the stream of hydrating medium includes an alkaline agent.

3. The process as defined in claim 1 in which the stream of hydrating medium comprises a solution of sodium carbonate.

4. The process as defined in claim 1 in which the stream of hydrating medium comprises a heated solution of soluble salt having no deleterious action upon the oil or soap stock.

5. The process as defined in claim 1 in which the hydrating medium is added in an amount between 1 and 10% of the oil.

6. A continuous process of refining glyceride fats and oils which comprises the steps of: continuously admixing a stream of oil with a stream of refining reagent of such a character and amount as to produce foots incapable of satisfactory centrifugal separation, continuously conditioning said foots for centrifugal separation by continuously introducing a stream of hydrating medium thereby to convert the foots from their centrifugally inseparable form into a centrifugally separable form and continuously centrifugally separating the said conditioned foots from the refined oil.

7. The process as defined in claim 6 in which the hydrating medium constitutes a weighting agent.

8. The process as defined in claim 6 in which the hydrating medium comprises an alkaline agent.

9. The process as defined in claim 6 in which the alkaline agent added during the process is several times greater than that theoretically required to neutralize the free fatty acids.

10. A continuous process of refining glyceride fats and oils which comprises the steps of: mixing with said oils a refining reagent which reacts with the impurities of the oil to form foots of a centrifugally inseparable nature, converting the foots from their centrifugally inseparable form to a centrifugally separable form by introducing to the mixture a hydrating medium prior to centrifugal separation and continuously centrifugally separating the conditioned foots from the refined oil.

11. The process as defined in claim 10 in which the separation is effected in the absence of such quantities of alcohol as would substantially completely dissolve said foots.

12. A process of refining glyceride oils and fats which comprises the steps of: mixing with the oils an alkaline reagent in an amount not substantially greater than that required to effect neutralization of the free fatty acids, thereby producing soap stock incapable of satisfactory centrifugal separation, conditioning said soap stock for said separation by the incorporation therewith of a hydrating medium and thereafter continuously centrifugally separating the conditioned soap stock from the refined oil.

13. The process as defined in claim 12 in which the alkaline refining reagent comprises caustic alkali.

14. The process as defined in claim 12 in which tht hydrating medium comprises alkaline material capable of effecting color reduction.

15. The process as defined in claim 12 in which the hydrating medium includes sodium carbonate.

16. A continuous process of refining glyceride fats and oils which comprises the steps of: mixing said oils with an alkaline reagent and a small amount of alcohol, continuously conditioning the resulting soap stock for centrifugal separation by introducing a stream of hydrating medium into said mixture and separating the thus conditioned soap stock from the refined oil in the absence of such quantities of alcohol as would substantially completely dissolve the soap component of the soap stock.

17. The process as defined in claim 16 in which a stream of the mixture is continuously admixed with a stream of hydrating medium.

18. The process of refining glyceride oils and fats which comprises the steps of: admixing an alkaline refining reagent with said oil of such character and amount as to produce a soap stock incapable of continuous centrifugal separation, continuously conditioning the soap stock for such separation by continuously adding a hydrating medium comprising sodium carbonate whereby to modify the said soap stock, subjecting the mixture to elevated temperatures sufficient to liquefy the thus modified soap stock and continuously centrifugally separating the soap stock from the refined oil in the absence of such quantities of alcohol as would substantially completely dissolve the said soap stock.

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