Jan. 28, 1941. B. CLAYTON 2,230,196

PROCESS OF TREATING SOAP STOCK

Filed Feb. 13, 1940 2 Sheets-Sheet 1

Fig. 1.

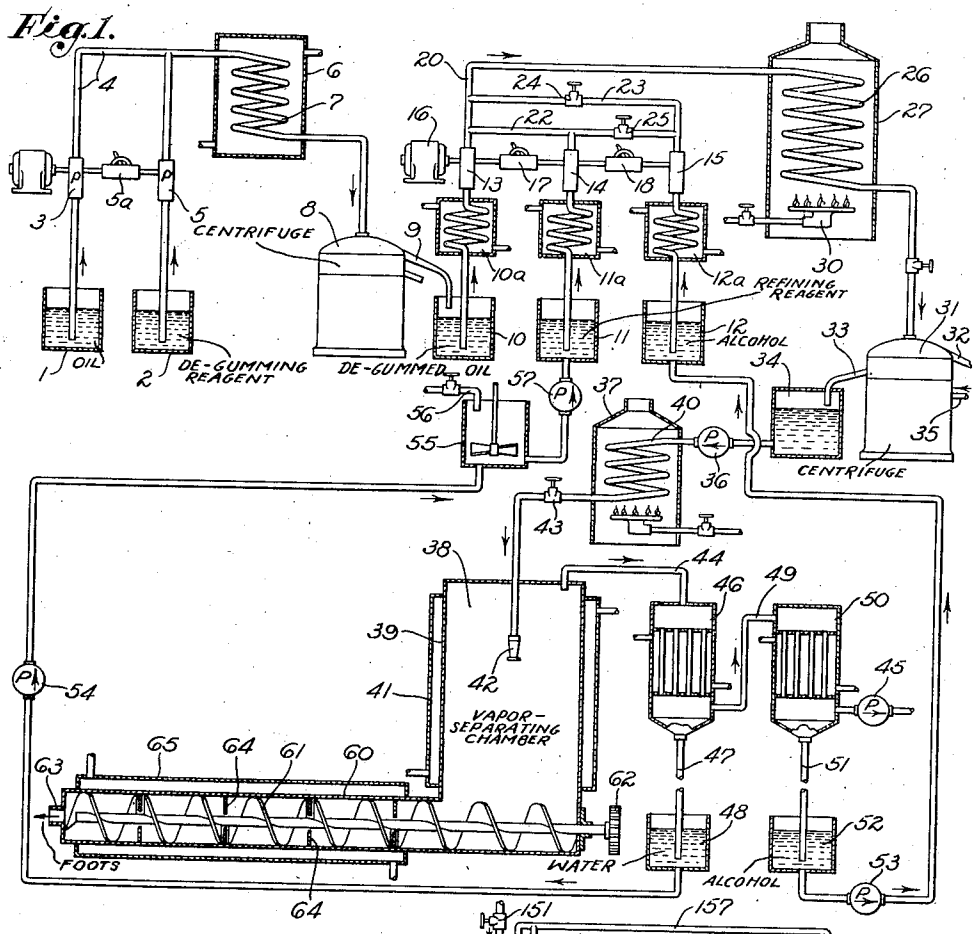
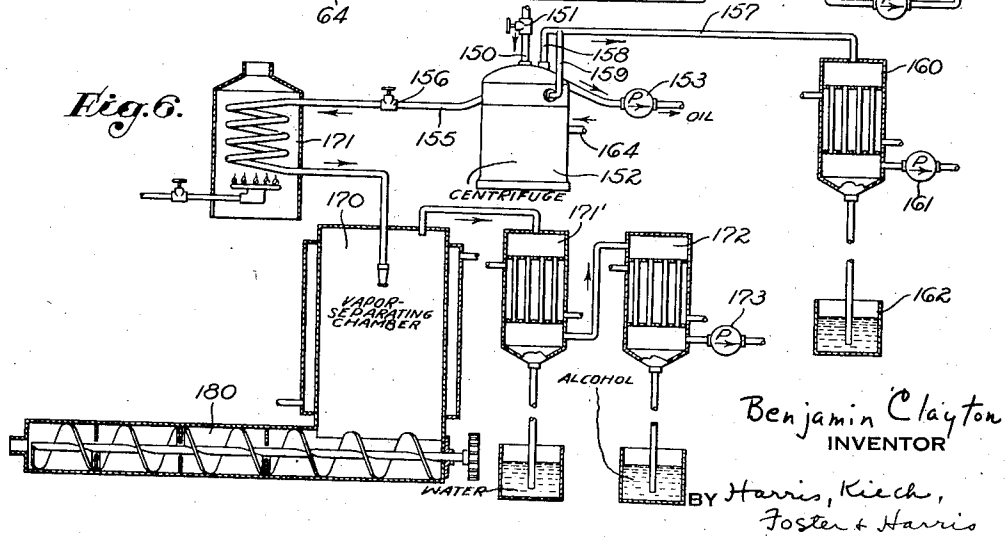



Fig. 6.




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Filed Feb. 13, 1940 2 Sheets-Sheet 2

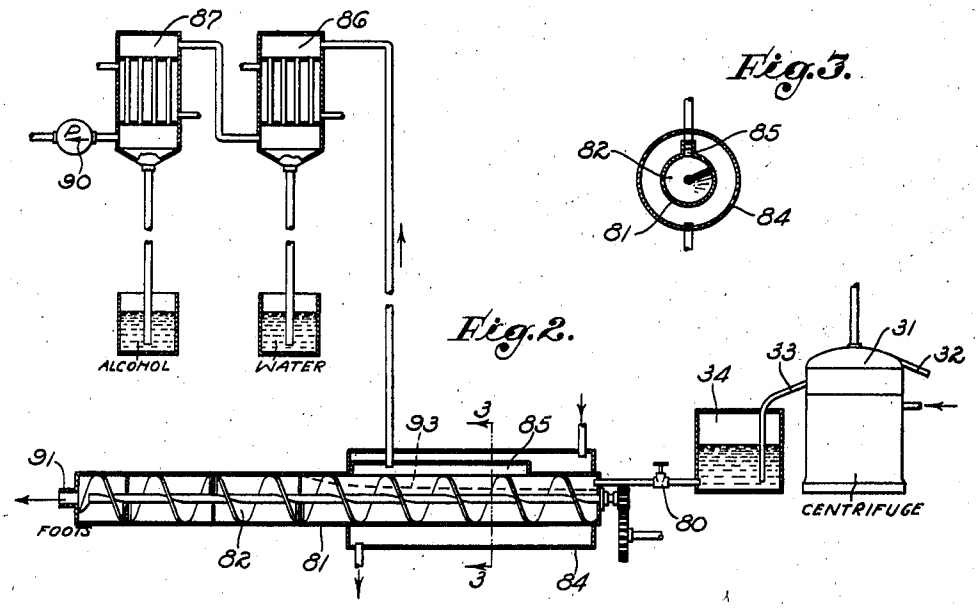



Fig. 2.




Fig. 3.

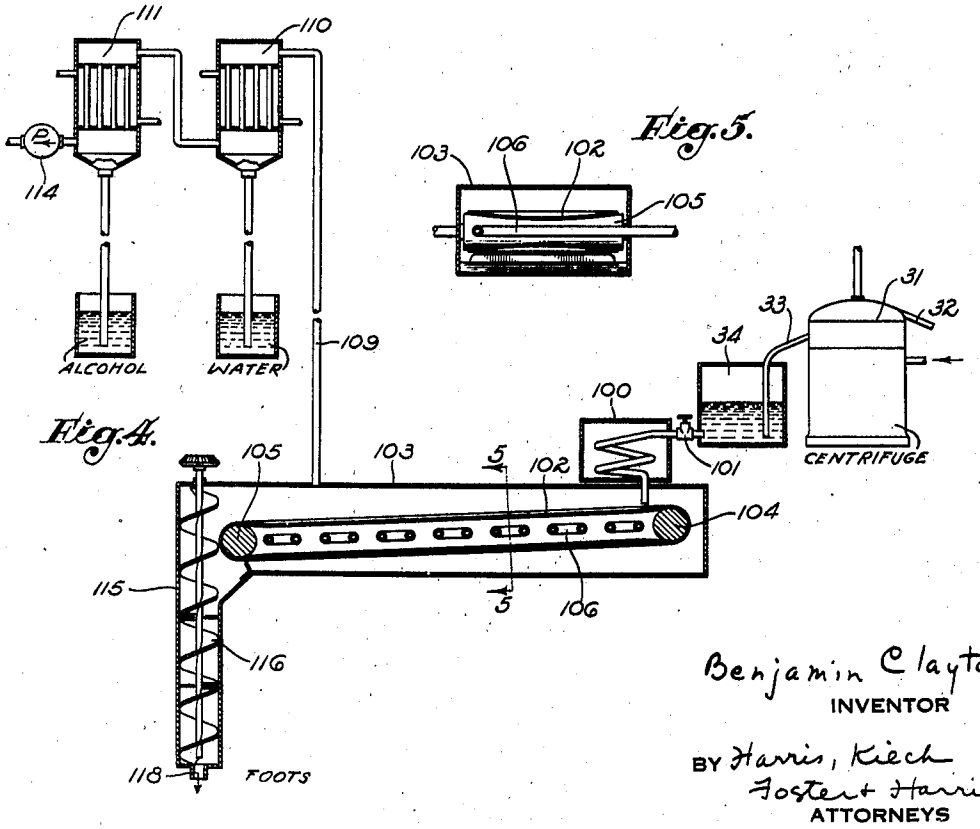



Fig. 4.




Fig. 5.

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Patented Jan. 28, 1941 2,230,196

# UNITED STATES PATENT OFFICE

2,230,196

## PROCESS OF TREATING SOAP STOCK

Benjamin Clayton, Houston, Tex., assignor to Refining, Inc., Reno, Nev., a corporation of Nevada

Application February 13, 1940, Serial No. 318,753

10 Claims. (Cl. 260—412.5)

My invention relates to a novel method and apparatus for refining glyceride-type oils, and has among its objects the lowering of refining losses without increasing the color of the refined oil, in many instances producing an oil of better color.

A crude glyceride-type oil contains free fatty acids and other impurities. For example, crude cottonseed oil may contain free fatty acids ranging in quantity from a fraction of one percent to more than two percent. It is common practice to refine such glyceride-type oils by use of an alkali refining agent, usually in dilute aqueous solution, such as caustic soda or caustic potash. These and other refining agents capable of reacting with the impurities to form foots and thus refining the oil are herein included in the term "refining reagent."

While such refining reagents have a preferential affinity for the free fatty acids, they also tend to react with the oil itself. The loss in volume of a crude glyceride-type oil during such refining is called the "refining loss." It is made up partly of the loss due to the formation of foots from the free fatty acids and other impurities initially in the oil, a necessary loss, and partly of the loss due to the action of the reagent on the oil itself and due to entrainment of oil in the separated foots, a loss which is very materially decreased in the present invention. It is almost invariably the practice to add a greater quantity of the refining reagent than is theoretically necessary to neutralize the free fatty acids, the excess acting to produce a proper bleach by removing objectionable color. However, in conventional batch treatment this excess tends to saponify a portion of the neutral oil, thus increasing the refining loss.

In reacting with the impurities, the refining reagent forms foots or soap stock. These foots are composed primarily of soap and various color impurities and are in the form of minute particles which tend to settle if allowed to stand quiescent. Oil may be associated therewith either by being emulsified within the particles, or by being entrained in the voids between such particles. Usually both types of association may coexist, especially if gravitational separation is utilized. In a continuous refining system utilizing a centrifuge for separating the oil and the foots it is possible to recover most of the oil in these voids, but the present invention goes even further in seeking to eliminate in addition losses due to the emulsified oil.

I have found it possible to decrease very materially saponification of neutral oil and reduce the amount of oil in the soap stock without increasing the color of the refined oil, even often producing an oil of better color, by proper use of a third material. This material is an agent which acts, in the quantities used, as a de-emulsifying agent and as an inhibitor for saponification of neutral oil, being hereinafter termed a "de-emulsifying and inhibiting agent." It is preferably volatile and has a tendency to break or prevent the formation of the usual uniformly emulsified character of the soap stock. Its power to inhibit the saponification of neutral oil is of particular importance as it reduces this saponification loss one-half or more. It may desirably be of such character as in effect to "grain out" color impurities.

Only sufficient quantity of this agent is used to inhibit saponification of neutral oil by the excess of the refining reagent employed, and to exert the desired de-emulsifying action. This quantity is preferably small, seldom exceeding more than a few percent and usually being between .75% and 1.7% based on the weight of the oil, though these limits are not invariable and may be departed from, being here set forth by way of illustration. With exceptional oils containing large amounts of fatty acids, for example, above 3%, it may be necessary to increase somewhat proportionally the quantity of the de-emulsifying and inhibiting agent. It is further desirable that such small quantity be used as not to interfere with satisfactory separation of the refined oil from the resulting soap stock.

The alcohols have been found very satisfactory as such a third agent, though various other chemical agents may be used. Monohydric or polyhydric alcohols may be used, but the former is preferred. For example, butyl, iso-propyl, propyl, amyl, ethyl or other monohydric alcohols give satisfactory results, those most miscible with water being preferred, for example, iso-propyl or ethyl. Iso-propyl alcohol has been found to be particularly advantageous. The term "alcohol" hereinafter used represents one example of a de-emulsifying agent and includes alcohols of the monohydric or polyhydric types, or mixtures thereof.

Various chemical agents other than alcohol can also be used as the de-emulsifying and inhibiting agent. Various of the esters can also be employed, for example, ethyl acetate, or the refining reagent may comprise sodium alcoholate, potassium alcoholate, etc.

The de-emulsifying and inhibiting agent may be added by mixing with the refining reagent, or it may be added to the oil before or after addition of this refining reagent. The first-mentioned system is preferred, the resulting refining solution usually containing from 10 to 25% of the alcohol or other de-emulsifying and inhibiting agent, though these limits can often be departed from. For instance, with grain alcohol it is possible to use even more alcohol than water in the solution. With iso-propyl alcohol it is usually desirable that the solution of alcohol and refining reagent contain not more than 15% thereof, though slightly more and up to 25% can be used.

It is an object of the present invention to reduce saponification of neutral oil in a batch or continuous process for refining glyceride-type oils, and to produce other desirable results in such a process.

It is another object of the present invention to refine glyceride-type oils by use of a refining reagent and an agent inhibiting saponification of neutral oil and acting as a de-emulsifying agent to reduce the amount of oil in the soap stock.

It is a further object of the invention to add and subsequently remove this de-emulsifying and inhibiting agent in such a process.

While batch operation of the invention is quite successful, I have found particularly good results and excellent economies in operating in a continuous manner. Any difficulty which may be experienced during batch treatment in uniformly mixing alcohol, for instance, with a large body of oil, can be overcome in a continuous process by mixing small quantities of the three materials, preferably by continuously mixing suitably proportioned streams thereof. If this mixing is done in a confined space there is no loss due to evaporation, and other very desirable ends are accomplished. The mixing action can be made very complete and the resulting mixture can be moved from the mixing zone as fast as it is formed.

It is an object of the present invention to add to one portion of a continuous refining system a de-emulsifying and inhibiting agent and to remove this agent at another portion of the system.

Another object is to mix small quantities of the materials, preferably by mixing proportioned streams thereof.

Another object of the invention is to move the resulting mixture from the mixing zone as fast as produced, preferably as a stream moving with such flow conditions that stratification or separation of the materials is prevented, thus insuring delivery of a uniform mixture to the separating means.

A proper excess of the refining reagent is preferable in the process over and above the amount theoretically required to combine with the free fatty acids, the excess being that customarily used and acting on the color impurities. The removal of such color impurities is a function of time, and while with certain oils this reaction is very rapid, some oils require a longer time of contact with this excess refining reagent. Heat may accelerate this color-removing reaction and facilitate later separation of the mixture. In the present invention flow of the mixture through an elongated zone will give this additional time, if required, and heat may be applied during this flow if desired.

It is an object of the present invention to mix a glyceride-type oil, a refining reagent and a de-emulsifying and inhibiting agent and condition the resulting mixture during flow through a passage before separating any of the materials in the mixture from the remaining materials thereof.

It is not difficult to separate the refined oil, the foots, and the de-emulsifying and inhibiting agent. Several alternatives can be used, and these products can be separated in various sequence. The resulting reaction products can be so treated as to first remove the de-emulsifying and inhibiting agent, for instance by vaporization, leaving a mixture of refined oil and foots which is later separated. On the other hand, the system herein described in detail first separates the refined oil from the resulting reaction products, subsequently separating the foots and the de-emulsifying and inhibiting agent.

It is an object of the present invention to separate the refined oil, foots, and de-emulsifying and inhibiting agent, regardless of the sequence of separation, and regardless of the expedients utilized to effect this separation, whether by vaporization, centrifugal action, gravitational action, filtration, etc.

Another object of the invention is to add a vaporizable agent to one portion of a refining system, then separate refined oil from the reaction products, and subsequently vaporize this agent from the foots.

While it is quite possible by use of the invention to refine some oils without use of temperatures above room temperature, refining is usually better at somewhat elevated temperatures. For instance, it is often possible to preheat the oil to a desired degree, and, if desired, the refining reagent and the de-emulsifying and inhibiting agent. In other instances, the mixture of these materials can be heated before separation. If centrifugal separation is used, it is often desirable to heat the zone where this centrifugal force is applied. Heat at this point is of material assistance in giving a uniform separation and insuring continuous operation over long periods of time, and this expedient can be used to the exclusion of heat applied before the mixture reaches the centrifuge.

It is an object of the invention to refine glyceride-type oils under controlled temperature conditions, either refining the oil in the cold, or applying heat at one or more positions in the system.

Another object of the invention is to provide a refining process capable of partially or completely dehydrating the foots by vaporization of water therefrom, if this expedient is desired.

In the preferred embodiment of the invention the dehydration of the foots may be accomplished under relatively high temperatures, and under pressure, if desired. I have found that if a mixture of foots and water, or a mixture of foots, water and a de-emulsifying and inhibiting agent, is subjected to temperatures from 212° to 600° F., the resulting dehydrated foots will have distinctly unexpected properties. Such treatment produces soap stock (foots) which is not of the odoriferous character produced by present processes. The high temperatures utilized apparently break down the odoriferous substances, usually proteins, into non-odoriferous products which are unobjectionable in the dehydrated soap stock. The resulting product can be used to make a better soap possessing none of the odoriferous

characteristics of present soaps made from conventional soap stocks.

The resulting soap stock will not ferment over long periods of time. Soap stock produced by conventional methods will ferment and necessitate shipping in steel tank cars. The improved product of the present invention can be shipped in any kind of container such as paper packages, wooden barrels, or loose in box cars, and will not ferment. It occupies less space and can be shipped in dust or powdered form. Its anhydrous character makes shipping quite economical as compared with ordinary soap stock which contains about 50% of water.

It is an object of the present invention to produce a dehydrated soap stock by subjection of the foots, usually associated with water or with water and a de-emulsifying and inhibiting agent, to a temperature sufficient to convert the odoriferous substances into non-odoriferous products, this temperature usually being between 212° and 600° F., and preferably between 300° and 600° F. The superior product can be obtained regardless of whether or not a de-emulsifying and inhibiting agent is present during such heating. In some instances, all or a portion of the water may be removed before subjection to high temperatures to convert the odoriferous substances, the provision of such a method being also within the objects of the present invention.

The new soap stock product is also a part of the present invention, being believed to be novel per se.

In some instances, the refining is made better by preliminarily treating the crude glyceride-type oil to remove gums and mucilaginous matter therefrom before the refining reagent acts upon the free fatty acids and other impurities to form the foots. It is an object of the present invention to preliminarily treat the incoming oil, if desired, to remove gums and mucilaginous matter therefrom. This often assists separation and further improves the color of the refined oil.

Further objects and advantages of the invention will be made evident hereinafter.

The drawings illustrate several embodiments of the invention. Referring particularly thereto:

Figure 1 represents a pipe line diagram of a refining system incorporating the features of the invention;

Figure 2 is a fragmentary view of an alternative system for separating the foots from the liquid substance;

Figure 3 is a section taken on the line 3—3 of Figure 2;

Figure 4 is a fragmentary view of another alternative system for separating the foots from the liquid substance;

Figure 5 is a section taken on the line 5—5 of Figure 4;

Figure 6 is a fragmentary view diagrammatically illustrating a system in which vaporization of the de-emulsifying and inhibiting agent may take place in part in the centrifuge.

Figure 1 diagrammatically shows a complete system capable of preliminarily treating the oil to remove gums and mucilaginous matter preparatory to refining. The particular de-gumming system diagrammatically shown in Figure 1 is very satisfactory in this regard but is not per se a part of the present invention, being claimed in the copending application of Benjamin H. Thurman, Serial No. 6,446, entitled "Method of treating vegetable oils and product obtained thereby." Various systems, either batch or continuous, may be used in this capacity, though the continuous system shown is particularly desirable in conjunction with the subsequent refining equipment.

As shown in Figure 1, the crude glyceride-type oil may be supplied to a tank 1, usually after preliminary filtration to remove fibrous materials or solids. The de-gumming reagent in tank 2 may comprise water, alcohol, or any electrolyte such as dilute aqueous solutions of acids, alkalies, or salts. A ten percent boric acid solution gives excellent results. This preliminary treatment, if used, is designed to remove gums and mucilaginous matter, not to react with the free fatty acids of the oil.

A pump 3 may continuously withdraw oil from the tank 1, delivering a stream thereof to pipe 4. A pump 5 may deliver a proportioned quantity of the de-gumming reagent to the pipe 4, being connected to the pump 3 by a variable speed mechanism 5a. These materials are intimately mixed together by any suitable mixing means, injection of one liquid into a stream of another having been found entirely satisfactory in this regard. The quantity of de-gumming reagent is preferably from 1 to 10 percent of the oil, by volume, though larger quantities of the de-gumming reagent may be used.

The mixture is heated in a heater 6 shown as including a coil 7, positioned in a container through which a suitable heating medium is circulated, the temperature being raised to a degree facilitating centrifugal separation when the mixture is introduced into a centrifuge 8. Here the reaction products are separated into de-gummed oil, delivered through a spout 9, and a material containing the gums, mucilaginous matter and other impurities of similar character. The de-gummed oil enters a tank 10 and may be withdrawn therefrom at a rate corresponding to the rate of introduction. It will be clear, however, that if this preliminary treatment is not desired, the crude glyceride-type oil can be moved directly into the tank 10.

A tank 11 may contain a body of the refining reagent, and the alcohol or other de-emulsifying and inhibiting agent may be retained by a tank 12. If desired, any or all of these three materials may be preliminarily heated in their respective tanks or may be heated in pre-heaters 10a, 11a, and 12a, before being mixed. Such preliminary heating is not necessarily used but offers one valuable way of controlling the temperature conditions of the system. If heat is applied to one or more of the three materials before mixing, particularly to the oil, it will facilitate pumping and may be used to increase the effectiveness of the later steps in the process.

By way of illustration, and for exemplary purpose only, the process will be described with reference to continuous operation, and with reference to alcohol as the de-emulsifying and inhibiting agent, the refining reagent being an aqueous alkali solution.

In a continuous process, it is desirable to mix the three materials in proportioned quantities in a confined space closed from the atmosphere. Preferably only small quantities of the materials are mixed at a time, and the mixture is preferably maintained with such flow conditions that the ingredients of the resulting mixture remain uniformly mixed to prevent premature separation. These ends are best accomplished by mixing proportioned streams containing these materials.

For example, proportioning pumps 13, 14 ar

15 may respectively draw these materials from the tanks 10, 11 and 12, these materials moving through the pre-heaters, if desired. These pumps may be driven by a drive means 16, preferably of the variable-speed type, and may be interconnected by variable-speed controls 17 and 18 which determine the relative proportions.

With these pumps properly adjusted to supply proportioned streams to the system, a resulting mixture is formed in a mixing zone which may comprise the forward part of a confined space defined in part by a pipe 20 into which the pump 13 may directly discharge.

Satisfactory mixing of the three materials can be obtained by suitable introduction of the alkali solution and the alcohol into the pipe 20, either separately or combined to form a refining solution. For instance, the pumps 14 and 15 may separately supply the alkali solution and the alcohol to the pipe 20 through pipes 22 and 23, the alcohol being supplied either upstream or downstream from the point of introduction of the alkali solution. On the other hand, it is often desirable to preliminarily mix the alkali solution and the alcohol to form a refining solution which is then introduced into the oil. This may be accomplished by closing a valve 24 in the pipe 23, and opening a valve 25 so that the pumps 14 and 15 discharge into the pipe 22, the resulting refining solution being then introduced into the pipe 20 to mix with the oil. In other instances, the alkali solution and the alcohol can be preliminarily mixed in the tank 11, the resulting refining solution being pumped through the pipe 22 into the pipe 20 in proportioned quantity.

Various auxiliary mixing devices such as a mechanical mixer can be used if desired, but a satisfactory mixing can be obtained by injection of one material into the stream of another. This minutely disperses the alkali and the alcohol in the oil, forming an excellent mixture and permitting the alkali to act very rapidly on the free fatty acids of the oil so that the resulting foots will be formed while the alcohol is present or immediately preceding the point of introduction of this alcohol. The resulting mixture will include oil, foots, water, alcohol, and any excess of the alkali over and above the amount theoretically necessary to combine with the free fatty acids, this excess acting upon the color impurities during continued flow through the pipe 20. By pumping the materials into such a confined space, the resulting mixture will move from the mixing zone as fast as it is formed. It is preferable to maintain such flow conditions in the confined space that stratification or premature separation of any of the ingredients is prevented.

Flow along a confined space such as is defined by the pipe 20 will give this desirable action, and will condition the mixture preparatory to separation. One phase of this conditioning involves action of the excess alkali on the color impurities. With some oils, this reaction is very rapid, in which event the confined space need not be necessarily long to give the desired reaction time. With other oils, a more prolonged time of contact is desirable, and a correspondingly longer confined space is desirable, thus increasing the reaction time before separation.

It is sometimes desirable to heat this resulting mixture during flow through such a confined space. In this capacity I have shown a coil 26 of a heater 27 as forming a part of the confined space, heat being supplied by any suitable means such as a burner 30. Other types of heaters providing a confined space closed from the atmosphere may be utilized, though in some instances no heat need be applied at this point. Flow therethrough should preferably maintain the uniformity of the mixture in the sense that stratification or premature separation is prevented, thus insuring delivery to a separating means of a mixture which does not vary from time to time in composition.

In the system shown, the resulting products are first treated in a manner to separate the refined oil. The separation is preferably continuous and Figure 1 illustrates a centrifuge 31 acting in this capacity. Conventional high-speed centrifuges can be used to separate the resulting products into refined oil and a liquid substance containing the foots, water, and the de-emulsifying and inhibiting agent, in this example alcohol. Thus, refined oil may be continuously discharged through a spout 32, and the liquid substance delivered through a spout 33 to a tank 34. Preferably such centrifuges will be substantially closed from the atmosphere to prevent loss of alcohol. However, superior separation, lower refining losses, and continuous operation over long periods of time can be effected by utilizing a heated centrifuge such as is disclosed in the application of Benjamin Clayton, filed August 1, 1935, Serial No. 34,258, now Patent No. 2,100,277, granted November 23, 1937. For instance, a heating medium such as steam may be supplied through a pipe 35 to the exterior of the rotating bowl of the centrifuge.

Refining of some oils can be accomplished by use of little or no excess temperature. For instance, the materials can be mixed at substantially room temperature and moved to the centrifuge 31 without intermediate heating, especially if the centrifuge is of the heated type. In most instances, however, superior results are obtained by pre-heating one or more of the incoming materials, or applying heat to the resulting mixture, or by using both of these expedients. Even then, however, better results will be obtained by use of a heated centrifuge. The desirable temperature of the resulting products issuing from the coil 26 will depend in part upon the type of separating means utilized and upon the character of the oil. If a centrifuge is used, very satisfactory results can be obtained by introducing the resulting products thereinto while at a temperature between 100° and 180° F. However, these temperature limits are not invariable, and temperatures somewhat above or below this range can often be used with success, depending upon the oil being refined, the type of centrifuge utilized, etc. A temperature between 120° and 140° F. is usually sufficient, especially if a heated centrifuge is used. For example, the mixed products may go to the heated centrifuge at 130° F., the refined oil discharging therefrom at substantially the same temperature, for instance 160° F., due to the heating of the bowl.

The tank 34 acts merely as a temporary receptacle, the liquid substance being preferably withdrawn therefrom as fast as introduced thereinto if continuous operation is desired. It may be vented to the atmosphere to maintain the pressure in the centrifuge 31 at substantially atmospheric value. In some instances this tank can be dispensed with.

The de-emulsifying and inhibiting agent, in this instance alcohol, may be separated from this liquid substance and from the foots by various

expedients. Vaporization is preferred, and three alternatives are indicated in Figures 1, 2 and 4.

In Figure 1 this liquid substance is continuously withdrawn from the tank **34** by a pump **36** or by a vacuum in the subsequent equipment. If desired, it may move through a heater **37** preparatory to introduction into a vaporizing chamber **38** defined by a container **39**. It is preferable that this heat be supplied during flow through a confined space such as is provided by the coil **40** shown. Heat may be supplied by a suitable burner or by a hot medium circulated exterior of such a coil. Sometimes very little, if any, heat need be applied at this point, and it is often possible to dispense with the heater **37**, moving the liquid substance directly into the chamber **38**. However, the temperature of the liquid substance prior to reaching the chamber **38**, or after it reaches this chamber, should be such that the de-emulsifying and inhibiting agent, in this instance alcohol, will assume a vapor state when sujected to the low pressure maintained in this vaporizing chamber **38**. This heat may be supplied by the heater **37**, by circulating a hot medium through a jacket **41** around the container **39**, or by injecting a hot medium such as steam into the chamber **38**. If desired, the flow of the liquid substance into the chamber **38** may be throttled as by a nozzle **42** or a valve **43**. Thus, pressures above atmospheric can be developed in the coil **40** if desired.

In this embodiment it is preferable to remove all of the alcohol by vaporization at this stage of the process. By proper control of temperature and pressure conditions it is also possible to remove all or a part of the water at this stage in the process, thus dehydrating the foots either partially or completely. By way of example, the system can be operated so that the liquid substance discharged from the centrifuge is at a temperature of about 160° F. If supplied at this temperature to the vaporizing zone **38** maintained under a sufficient vacuum, all of the alcohol and a part of the water will vaporize. If further heated in the heater **37**, or by the jacket **41**, or by supplying a heating medium to the chamber **38**, all of the water can be vaporized and removed along with the alcohol vapors.

The resulting vapors may be forcibly withdrawn through a pipe **44** by use of a vacuum pump **45**. If vapors of both water and alcohol are withdrawn, they may be fractionally condensed or condensed together and later separated by any means well known in the art of separating such materials. Fractional condensation is illustrated in Figure 1, the water being condensed in a condenser **46** and dropping through a barometric column **47** to a tank **48**, the alcohol vapors moving through a pipe **49** and being condensed in an alcohol condenser **50**, the condensate moving downward in a barometic column **51** to a tank **52**. These condensers may be of the tube type, the tubes being surrounded by a circulating cooling medium. The lower ends of the barometric columns may be submerged, and these columns are of sufficient height to balance the low pressure developed by the vacuum pump **45**.

The alcohol can be recovered almost entirely and is preferably re-used in the system. A pump **53** may withdraw this alcohol from the tank **52** and deliver it to the tank **12**. Similarly, the water may be re-used, being withdrawn from the tank **48** by a pump **54** discharging into a mixing tank **55**. Here the refining reagent may be prepared, as by mixing alkali therewith, this alkali being delivered through a pipe **56**. The resulting alkali solution is moved by a pump **57** into the tank **11**.

The foots which remain after vaporization of the alcohol are preferably withdrawn continuous from the chamber **38** by any suitable pumping means capable of withdrawing these foots without impairing the vacuum in this chamber. Figure 1 discloses a screw conveyor acting in this capacity. This conveyor may provide a housing **60** opening on the chamber **38**, and a screw **61** rotating therein under the influence of a drive means **62**. The foots are thus advanced leftward inside the housing **60** and the pressure progressively increases therein until sufficient pressure is developed to extrude the foots through an extrusion means **63**. This conveyor may thus act as a pump, intaking at the low pressure maintained in the chamber **38** and developing a sufficient pressure to extrude the foots. It acts as a vacuum seal and withdraws the foots without imparing the low pressure in the chamber **38**. The screw **60** is preferably formed in flights, with stationary members **64** extending inward from the housing **60** between these flights to better insure continuous movement of the foots.

It is often desirable to subject the foots or soap stock to high temperatures to produce the improved product hereinbefore mentioned. In Figure 1, this can be accomplished by applying sufficient heat either in the heater **37** or in the chamber **38**, preferably the former. Thus, sufficient heat can be supplied in the coil **40** over and above that required to vaporize the alcohol. The liquid substance can be raised to a temperature between 212° and 600° F. to convert the odoriferous substances in the soap stock into non-odoriferous products. Most satisfactory operation is obtained by use of temperatures between 300° and 600° F. if this product is to be obtained, though the temperature will vary with different soap stocks. Even higher temperatures can be utilized in some instances without departing from the spirit of the invention, it being clear that there is no possibility of injuring the refined oil, for this oil has previously been separated. This heating may be carried out under pressure by partially closing the valve **43** or properly designing the nozzle **42**. However, this is not absolutely necessary.

If this non-odoriferous soap stock is to be produced by use of high temperatures, it is often possible to dispense with the use of any high vacuum in the chamber **38**, for the heat applied before or after reaching this chamber may be sufficient to vaporize the alcohol and substantially all of the water even in the absence of a high vacuum.

On the other hand, in making this non-odoriferous soap stock it is sometimes desirable to first extract the water and subsequently heat the soap stock to the temperature which will convert the odoriferous substances. For instance, it is possible to use temperatures in the coil **40** or in the chamber **38** only sufficient to remove the alcohol and most of the water, preferably using a high vacuum in this chamber. The resulting dehydrated soap stock may later be subjected to higher temperatures. For instance, it can be heated during the movement through the conveyor housing **60** by applying hot products of combustion to the exterior of this housing, or by circulating a hot medium through a jacket **65** therearound. In any of the alternatives hereinbefore mentioned, the soap stock can be cooled before exposure to the atmosphere, if desired.

It will be clear that the production of this non-odoriferous soap stock is not essential to that concept of the invention relating to the use of a de-emulsifying and inhibiting agent in conjunction with the refining of the oil. If soap stock of this character is not desired, it is entirely possible to use lower temperatures in removing the water and alcohol, for instance. In such case, it is often possible to dispense with all heating immediately before or after the liquid substance enters the chamber 38. On the other hand, if this non-odoriferous soap stock is to be produced, it will be clear that use of a de-emulsifying and inhibiting agent such as alcohol is not absolutely essential in the refining step. While a very desirable product of non-odoriferous character can be thus obtained, soap stock derived from other refining systems not necessarily using a de-emulsifying and inhibiting agent can be rendered non-odoriferous by subjection to the temperatures above mentioned.

Figure 2 shows a system in which the alcohol or other de-emulsifying and inhibiting agent, and if desired, all or a portion of the water, may be vaporized during flow of the liquid substance through a conveyor. Here, the liquid substance flows from the tank 34, or alternatively direct from the centrifuge, through a valve 80 and enters a conveyor housing 81 in which a screw 82 rotates. The entrance portion of the conveyor housing is surrounded by a jacket 84 through which a suitable heating medium is circulated to raise the temperature of the liquid, if not already high enough, to such an extent that the alcohol or other de-emulsifying and inhibiting agent is vaporized as well as any desired portion of the water if desired.

The vapors can be removed through a manifold 85, being discharged into a condenser means shown as including condensers 86 and 87 similar to the condensers 46 and 50, previously described.

While vaporization at atmospheric pressure may be used, it is often desirable to decrease the pressure in the entrance end of the conveyor housing to permit removal of the vapors at lower temperature. For instance, a vacuum pump 90 may be connected to the condenser 87 to lower the pressure in the entrance portion of the conveyor housing 81. The material entering the conveyor housing through the valve 80 is a liquid. As the alcohol is vaporized the foots remaining in the advancing stream become thicker. If sufficient heat is supplied to vaporize the water in whole or in part, as well as the alcohol, these foots will be partially or completely dehydrated and will assume the form of a compacted mass which is moved leftward by the conveyor and extruded through an orifice 91, thus sealing the vacuum. Thus, regardless of whether the water is vaporized, the surface of the material in the conveyor housing may be approximately as indicated by the dotted line 93, only the left-half end of the conveyor being completely filled with the foots.

In Figure 4 another alternative is shown. The liquid substance moves from the tank 34 through a heater 100 as controlled by a valve 101, being delivered to a belt conveyor 102 positioned in a housing 103. The belt may be of the endless type extending around rollers 104 and 105, suitably driven. This belt may be of the type which assumes a trough-like form in the space between the rolls to better retain the liquid substance delivered thereto by the heater 100.

The liquid substance may be further heated during the time it is being advanced by the belt 102. Figure 4 illustrates by way of example a series of pipes 106 extending through the zone between the upper and lower portions of the belt, the heating medium being circulated therethrough to heat the liquid substance sufficiently to drive off the alcohol or other de-emulsifying agent in vapor form if it was not previously at such temperature. The vapors move from the housing 103 through a pipe 109 to condensers 110 and 111 similar to those previously described. If evaporation under low pressure is desired, a vacuum pump 114 may be connected to the condenser 111 to maintain a partial vacuum in the housing 103. Application of sufficient heat, or utilization of sufficiently low pressure, or both, may be used to partially or completely dehydrate the foots during this forward movement, if desired.

The foots, either containing water or in substantially anhydrous form, are discharged from the belt 102 near the roller 105 and drop into a suitable pump capable of continuously withdrawing the foots without impairing any low pressure which may be used in the housing 103. A screw conveyor is shown in this capacity, providing a housing 115 with a screw 116 rotating therein. If low pressure is used in the housing 103, this conveyor will progressively increase the pressure on the foots, ultimately extruding these foots through an orifice 118.

Figure 6 diagrammatically illustrates an alternative system which will also be described with reference to alcohol as the de-emulsifying and inhibiting agent. The first part of the system is the same as shown in Figure 1, the reaction products flowing from the heater 27 through a pipe 150 as controlled by a valve 151 moving to a centrifuge 152 which is maintained under a partial vacuum. Centrifuges of this type are known in the art, and the refined oil can be forcibly withdrawn by a pump 153, the liquid substance containing foots, water and usually a portion of the alcohol, being continuously withdrawn through a pipe 155 provided with a valve 156. The temperature of the incoming reaction products may be such that a portion of the alcohol will vaporize in the centrifuge 152, the vapors being withdrawn through a pipe 157 with branch pipes 158 and 159 respectively communicating with the upper and lower pans which collect the oil and the foots. These vapors may be condensed in a condenser 160, being drawn thereinto under the influence of a vacuum pump 161. The condensate drops through a barometric column to a tank 162.

Liberation of vapors in the centrifuge is facilitated by supplying a heating medium through a pipe 164, the centrifuge being of the heated type previously mentioned.

If desired, substantially all of the alcohol can be removed at this stage of the process, though it is preferable to so operate the system that only a portion thereof separates at this point. The remaining alcohol, and any desired portion of the water, can be removed by moving the liquid substance directly into a low-pressure zone 170, or preliminarily through a heater 171 corresponding to the heater 37 previously mentioned. A higher vacuum may be maintained in the chamber 170 than in the centrifuge 152, if desired. The remaining alcohol, and the water if desired, can be removed from this chamber 170 in vapor state to condensers 171' and 172 under the action of a vacuum pump 173.

The foots either containing substantially all of the water or partially or completely dehydrated, are continuously withdrawn and extruded by means of a conveyor 180 similar to the conveyors previously mentioned.

In conclusion, the following example will indicate the superior results obtainable by the use of a de-emulsifying and inhibiting agent used in conjunction with a refining reagent, considered with reference to production of a refined oil in the system shown in Figure 1 and using a crude cottonseed oil, not previously de-gummed, and containing 1.4% free fatty acids. Using no alcohol and 4.44% of 20° Bé. refining reagent, there was a loss due to excess saponification of 3.37%, the resulting refined oil having a color of 35 yellow, 6.0 red. When the same percentage of refining reagent was mixed with 1.1% isopropyl alcohol to form a refining solution, and this was mixed with this crude cottonseed oil, the excess saponification was reduced to 2.29%, effecting a saving of more than 1%. The color of the refined oil when using the alcohol was 35 yellow, 5.9 red. Other comparative runs show even better results and definitely establish the superiority of the present invention in inhibiting saponification of neutral oil, reducing the amount of oil in the soap stock, and often producing better colors evident either from the refined oil before bleaching or from this oil after washing, drying and bleaching by conventional methods.

It will be understood that while the embodiments herein illustrated relate to a continuous process, this mode of operation is not essential. Batch operation is possible, the de-emulsifying and inhibiting agent being added to the kettle, preferably closed and communicating with a condenser means for recovering this agent, for instance alcohol. The resulting mixture of refined oil, foots and the de-emulsifying and inhibiting agent can be separated in the kettle, the refined oil being separately withdrawn. The remaining liqu'd substance can be treated as hereinbefore described to separate the de-emulsifying and inhibiting agent by vaporization thereof or by other expedients. On the other hand, the resulting mixture can be withdrawn from the kettle and the ingredients separated by various means and in various sequence.

I claim:

1. The process of purifying soap stock from the alkali refining of glyceride oils, which comprises, heating said soap stock to a temperature sufficiently high to break down odoriferous substances contained therein and removing vaporizable materials from said soap stock in vapor form to produce an improved soap stock.

2. The process of purifying soap stock containing water and odoriferous substances, which comprises, heating said soap stock to a temperature sufficiently high to break down said odoriferous substances and vaporizing water and other vaporizable materials from said soap stock.

3. The process of purifying soap stock from the alkali refining of glyceride oils, which comprises, continuously heating a stream of said soap stock to a temperature sufficiently high to break down odoriferous substances contained therein, discharging the resulting heated stream of soap stock into a vapor separating chamber and removing vaporizable materials from said soap stock in said chamber.

4. The process of purifying soap stock from the alkali refining of glyceride oils, which comprises, heating a flowing stream of said soap stock under pressure and out of contact with the atmosphere to a temperature sufficiently high to break down odoriferous substances contained therein, discharging the resulting heated stream of soap stock into a vapor separating chamber, withdrawing vaporizable materials in vapor form from said chamber at a rate sufficient to maintain a vacuum therein.

5. The process of purifying soap stock from the alkali refining of glyceride oils, which comprises, heating a flowing stream of said soap stock under pressure and out of contact with the atmosphere to a temperature sufficiently high to break down odoriferous substances contained therein, discharging the resulting heated stream of soap stock into a vapor separating chamber, withdrawing vaporizable materials in vapor form from said chamber at a rate sufficient to maintain a vacuum therein, and continuously removing the purified soap stock from said chamber.

6. The process of purifying soap stock from the alkali refining of glyceride oils, said soap stock containing water, a lower aliphatic alcohol and odoriferous substances from said oil, which comprises, heating said soap stock to a temperature sufficiently high to break down odoriferous substances and removing vaporizable materials including said water and alcohol from said soap stock as vapors in order to produce an improved soap stock.

7. The process of dehydrating soap stock from the alkali refining of glyceride oils, which comprises, continuously heating a stream of said soap stock to a temperature sufficiently high to vaporize water contained in said soap stock when the same is discharged into a vapor separating zone, discharging the heated stream of said soap stock into a vapor separating zone and continuously removing water in vapor form from said vapor separating zone.

8. The process of purifying soap stock from the alkali refining of glyceride oils, which comprises, heating said soap stock during continuous movement thereof to a temperature sufficiently high to break down odoriferous substances contained therein, and removing vaporizable materials from said soap stock as vapors to produce an improved soap stock.

9. The process of purifying soap stock from the alkali refining of glyceride oils, which comprises, heating said soap stock to a temperature ranging from 212 to 600° F. and sufficiently high to break down odoriferous substances contained therein and removing vaporizable materials from said soap stock in vapor form to produce an improved soap stock.

10. The process of purifying soap stock from the alkali refining of glyceride oils, which comprises, heating said soap stock to a temperature ranging from 300 to 600° F. and sufficiently high to break down odoriferous substances contained therein and removing vaporizable materials from said soap stock in vapor form to produce an improved soap stock.

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