

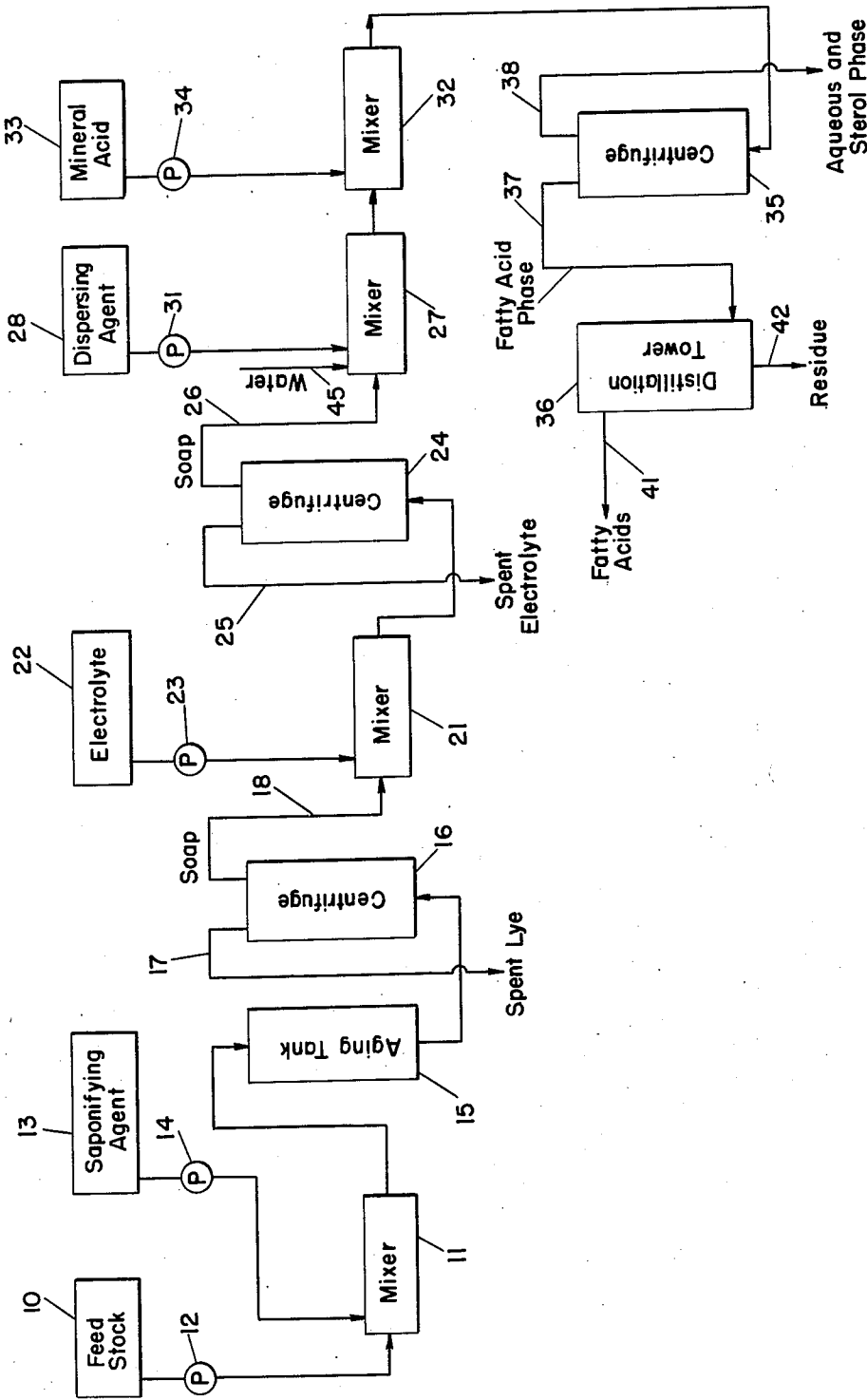
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PRODUCTION OF FATTY ACIDS FROM SOAPS

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PRODUCTION OF FATTY ACIDS FROM SOAPS

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13 Claims. (Cl. 260—413)

This invention pertains generally to the production of higher fatty acids, and pertains particularly to the production of such acids from glyceride oils and fats.

In the production of higher fatty acids from glyceride oils and fats (hereinafter referred to for convenience collectively as "oils"), it is known to treat such oils with an aqueous solution of an hydroxide of an alkali metal or alkaline earth, or mixture thereof, to saponify such oils, and to simultaneously wash the saponified mass one or more times with an aqueous electrolyte solution to remove impurities. This is followed by the treatment of the washed saponified mass with an aqueous solution of a strong mineral acid, e. g., sulfuric acid or hydrochloric acid, called "acidulation," to displace the fatty acids from their salts which comprise the soap, whereupon after separation of the aqueous phase from the non-aqueous phase thus formed and containing the freed fatty acids, the non-aqueous phase is subjected to distillation for the purification and recovery of fatty acids.

Typical saponifying agents are sodium hydroxide and potassium hydroxide, sodium hydroxide being more commonly employed.

Examples of hydroxides of the alkaline earths are calcium hydroxide and magnesium hydroxide.

The electrolyte solutions employed for washing purposes may be aqueous solutions of the hydroxides, and/or of the mineral acid salts thereof, and usually containing the same cation as the hydroxide employed for saponification purposes. Of the hydroxides, sodium hydroxide is more commonly used, and of the salts, sodium chloride is more commonly used. Usually the concentration of the electrolyte in the aqueous solution is such that the solution is capable of dissolving very little, if any, soap. This results in what is generally known as "graining" or "opening" of the soap. While the soap may be "closed" between successive washings by the addition of water thereto, this is by no means necessary for effective washing of the soap.

While the oils to be saponified may be derived from any source, it is more common to start with the material called "soapstock" which is a by-product obtained in well-known methods of refining glyceride oils (including fats) with alkaline reagents, e. g., sodium hydroxide and/or sodium carbonate. Such refining operations have for their purpose the neutralization of the free fatty acids present in the oil, and/or the removal of color and/or gums or other impurities from the oil. Some of the oil may be saponified during such treatment, although this is usually held to a minimum. As a result of the refining treatment, soap is formed by virtue of the neutralization of free fatty acids and not infrequently to some extent by virtue of saponification of oil, which soap must be removed from the treated oil, e. g., by gravity settling or centrifuging. However, irrespective of the procedure employed for separation of the soap from the oil, some oil invariably is left behind in the separated soap which as indicated above is known in the trade as "soapstock."

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Examples of glyceride oils are cottonseed oil, sesame oil, sunflower seed oil, corn oil, soya bean oil, coconut oil, palm oil, palm kernel oil, peanut oil, rape seed oil, babasu oil, linseed oil, tallow, lard, grease, fish oil, whale oil, etc., i. e., any of the glyceride oils and fats whether of vegetable or animal origin.

When soapstock is employed for higher fatty acid production, it is known to subject it to saponification conditions, usually with an hydroxide containing the same cation as the reagent employed for the refining of the glyceride oil, to saponify the neutral oil contained therein, and to follow this by the washing, acidulation and distillation referred to above.

In the refining of glyceride oils, gums are sometimes separated independently of the soapstock by a procedure known in the art as "degumming." The separated gums invariably contain a considerable percentage of glyceride oil, and thus comprise another excellent source of raw material for the production of fatty acids following in general the procedure applied to other sources of glyceride oil.

In the production of fatty acids by the above-outlined procedure, it has heretofore been necessary to operate by batch methods, and no continuous method, insofar as I am aware, has heretofore been perfected. An indeterminate time factor, running into many hours of boiling or other manipulation, has heretofore been found necessary to effect satisfactory acidulation of the soap, and particularly soapstock, with the mineral acid, and separation into phases, requiring treatment thereof in a kettle or other large container. An example of the time required in the case of soapstock is sixteen hours for steam blowing and settling.

I have discovered that by the introduction of a small amount of a dispersing agent which is effective in mineral acid media into the acidulation zone, the action of the aqueous mineral acid upon the soap is accelerated to the point where continuous processing becomes feasible. Moreover, as the result of the presence of such a dispersing agent, a sharp separation into phases occurs very rapidly during the course of the acidulation. The action of the mineral acid on the soap is to displace the fatty acids therefrom with the formation of a fatty acid phase separate from an aqueous mineral acid phase. At the same time, and particularly in the case of soapstock, a phase usually of intermediate density between the fatty acid phase and the aqueous phase is formed, and which in the case of soapstock contains, among other substances, glucoside sterols.

A surprising feature of my invention is that a reagent having dispersing properties acts not only to greatly speed up the action of the aqueous mineral acid on the soap to displace the fatty acids, but also to greatly speed up the formation distinct from each other of the resulting phases. My invention lends itself to continuous separation either by gravity settling, or by centrifuging. It is to be understood, however, that outstanding advantages result even when my invention is applied to prior art batch methods, in that it greatly speeds up the acidulation and separation of the resulting phases in a kettle or other container employed in such methods.

When employing gravity settling, the phases may be separated from each other in any desired manner, e. g., by drawing off or decantation, as a whole or intermittently or continuously, as desired.

When three phases are present, such as in the case of soapstock, and separation is by centrifuging into two streams, it is a simple matter to remove the phase of intermediate density from the centrifuging zone, either with the aqueous phase or with the fatty acid phase, by mere adjustment of hydrostatic balance as is well understood

in the centrifuging art. For example, in the case of a centrifuge having a rotor of the open bowl type, this merely requires an adjustment of the radial distance from the axis of rotation of the discharge weir, e. g., ring dam, controlling the discharge of the heavier phase. In the case of a centrifuge having a rotor of the full bowl type, i. e., one which operates with the rotor completely full of liquid, this merely requires a control of the back pressure on one of the two discharging streams. If desired, a centrifuge capable of discharging each phase separately may be employed. Usually I prefer to discharge the intermediate phase when present along with the aqueous phase, and to recover the fatty acid phase separately from the other two phases. While the dispersing agent may be added to the zone of acidulation at any point prior to separation of the phases, since the dispersing agent acts to disperse the soap mass, I prefer to add the dispersing agent to the soap mass prior to or during its contact with the aqueous mineral acid reagent.

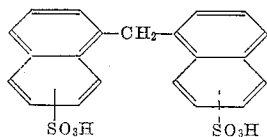
Any technique known in the art may be employed for effecting the acidulation, and for the subsequent distillation, all of which are well known to persons skilled in the art. For example, in carrying out the acidulation, a sufficient quantity of mineral acid, e. g., sulfuric acid or hydrochloric acid, is added to the acidulation zone to effect the desired displacement of the fatty acids from the soap. A pH of between 0 and 3 in the aqueous phase is illustrative, although any other desired pH may be employed.

Any dispersing agent for the soap which is effective in the acidulation zone, i. e., in the presence of strong mineral acid, may be employed without limitation. Preferably, however, I employ a dispersing agent which does not cause a large degree of foaming in the acidulation zone, since foaming in no way assists in the practice of my invention, and a large degree of foaming may cause inconveniences in the manipulation of the acidulated mass. Should a large degree of foaming result and it is desired to suppress the same, the use of a small amount of an anti-foaming agent, such as glycol stearate, octanol-2, 2-ethylhexanol, cyclohexanol, lauryl alcohol, cetyl alcohol, ethyl oleate, castor oil, the higher 1,2 and 1,3 glycols, etc. is recommended, though this is not essential.

The use of large excesses of a non-foaming type dispersing agent may result in the production of foam, which can be avoided either by adding an anti-foaming agent, but preferably and more economically, merely by reducing its proportion, the use of excesses obviously being wasteful.

Outstanding agents for dispersing the soap are as follows:

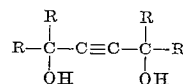
1. The naphthalene sulfonates in which two or more naphthalene nuclei are joined by alkylene groups. The prototype of this class is dinaphthylmethanemonosulfonic acid, the disulfonic acid having a formula as follows:



Products of this class are of indefinite composition. They may be manufactured by heating naphthalene, formaldehyde, and sulfuric acid together, or by treating naphthalene sulfonic acids with formaldehyde. Thus three or more naphthalene nuclei may be joined together by alkylene groups to yield a condensation polymer. Lower alkylated naphthalenes may also be used in the reaction. An example is monoisopropyl naphthalene. In place of naphthalene, other aromatic hydrocarbons may be employed such as benzene, diphenyl, anthracene, phenanthrene, fluorene, etc. or homologues or derivatives thereof. The salts of the foregoing acids, such as the sodium salts possess similar properties. The production of dispersing agents of this type is described in detail in the literature

and in prior art patents, and representative compounds are available in the trade under the trademarks Tamol, Leukanol and Daxad.

2. The tertiary acetylenic glycols representative of which are compounds having the formula



in which each R is an alkyl group. Examples of such compounds are 2,5-dimethyl-3-hexyne-2,5-diol, and 3,6-dimethyl-4-octyne-3,6-diol.

3. Quebracho extract.

4. Sodium hexametaphosphate type dispersing agents. Examples are the products available in the trade under the trademarks Calgon, Giltex, Quadrafos, and Hagan phosphate, and which contain sodium hexametaphosphate as the principal ingredient.

5. Dispersing agents comprising lignin derivatives, and produced principally from waste sulfite liquor by processes well known in the art, such as liginosulfonic acid compounds and liginosulfonate compounds, representative of which are sodium liginosulfonate, and calcium liginosulfonate. Products of this character are available in the trade under the trademarks Marasperse, Marasperse C, Marasperse CB and Marasperse N.

Further features of the invention will become apparent to persons skilled in the art as the specification proceeds, and upon reference to the drawing, in which

The single figure shows a flow diagram illustrating a procedure for the production of fatty acids in which my invention is embodied.

On the flow sheet at 10 is shown a container for feed stock to be subjected to the initial step of saponification. As above pointed out this feed stock may be comprised of glyceride oil or fat from any source, soap containing residual glyceride oil or fat (i. e., soapstock), or gums containing such residual oil or fat, and obtained as a by-product in the refining of such glyceride oil or fat, being a source of feed stock to which my invention is particularly applicable.

Feed stock from container 10 is fed to mixer 11 by means of pump 12 along with an aqueous saponifying agent, e. g., aqueous sodium hydroxide, from container 13 fed by means of pump 14.

Saponification of the feed stock by virtue of the presence of the saponifying agent takes place in mixer 11, and in the procedure undergoing illustration is brought to substantial completion therein. 12 and 14 preferably are proportioning pumps, and it is convenient, though not necessary, to have the proportion of saponifying agent to feed stock such as to provide a sufficient stoichiometric excess of saponifying agent to available unsaponified oil in the feed stock to cause eventual graining of the soap present as the mixture approaches equilibrium conditions. However, it is to be understood that aqueous electrolyte for the purpose may be otherwise supplied. As described and claimed in copending application Serial No. 448,867, filed August 10, 1954, by Vincent G. Bell, Jr. and Frederick W. Keith, Jr., when the feed stock is a by-product of the refining of glyceride oil, which term includes fats, a time factor is involved in obtaining a grained soap mixture. This time factor becomes greater when the stoichiometric excess of sodium hydroxide, or the percentage of electrolyte otherwise supplied, does not substantially exceed that required, after saponification is complete, to yield an aqueous electrolyte of a concentration just sufficient to grain the soap, or, in other words, of a concentration below which the aqueous electrolyte solution would begin dissolving increasing quantities of soap, and referred to herein for convenience as the "critical concentration." This time factor may be very substantially reduced, making continuous operation possible, by maintaining the mass, after the saponification treatment, rela-

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tively quiescent. To provide both the time factor and the relative quiescence for continuous operation, tank 15 is employed into which the mixture from mixer 11 is delivered, and down through which the mixture slowly descends.

The residence time of the mixture in tank 15, as it flows downwardly therethrough, is sufficient to afford the desired graining of the soap with consequent washing of impurities therefrom by the aqueous electrolyte solution.

It will be understood that the required residence time in aging tank 15 will vary between feed stocks, and that, generally speaking, it will decrease with increase in concentration of electrolyte in the aqueous solution employed for graining (i. e., washing) purposes. A longer residence time for the mixture in mixer 11 does not result in the desired graining, and graining is greatly accelerated by the avoidance, preferably insofar as possible, of mechanical agitation after saponification, and this is very effectively accomplished by the use of tank 15 as described.

The grained mixture from tank 15 is fed to centrifuge 16 in which the aqueous phase called "spent lye" is separated from the soap, the spent lye being taken off through line 17, and the soap through line 18. The soap is delivered by line 18 to mixer 21, wherein an additional washing of the soap with aqueous electrolyte solution takes place.

To effect washing in mixer 21, aqueous electrolyte solution, e. g., aqueous sodium chloride and/or sodium hydroxide, is delivered thereto from tank 22 by means of pump 23 which may be a proportioning pump. On the other hand, the flow may be by gravity, in which case 23 may be a flow meter. Any other means for feeding electrolyte solution may be provided.

The electrolyte in the aqueous solution fed to mixer 21 from tank 22 may be of any desired concentration. Preferably it is at least such that no significant quantity of soap is dissolved therein at equilibrium. Similar to the case of the electrolyte solution employed for the first graining or washing, the concentration of electrolyte in the aqueous phase in mixer 21 at equilibrium may be close to the so-called "critical concentration," such as slightly above. It is to be understood, however, that any desired concentration of electrolyte may be employed.

The mixture from mixer 21 is fed to centrifuge 24 in which the aqueous phase is separated from the soap, the aqueous phase, called "spent electrolyte," being taken off through line 25, and the soap through line 26, the latter leading to mixer 27.

If desired, the elements 21 to 26 may be omitted, whereupon the soap from line 18 would be fed directly into mixer 27, or the elements 21 to 26 may be duplicated any desired number of times, if it is desired, to provide one or more additional washing stages connected in the series.

In mixer 27 the soap is mixed with a small amount of dispersing agent effective in mineral acid media which for feed purposes conveniently is aqueous. As illustrated, an aqueous solution of dispersing agent is fed to mixer 27 from tank 28 by means of pump 31 which may be a proportioning pump, although it will be understood that any other means for feeding dispersing agent to mixer 27 may be substituted, e. g., the feed may be by gravity, and 31 may be a flow meter.

I prefer to feed the dispersing agent into mixer 27 in the form of an aqueous solution because the water thus added assists in the rapid incorporation of dispersing agent in the soap. I prefer to convert the soap from a grained condition into one having a smooth texture comparable to that of "closed" soap, and sufficient water may be added from tank 28 along with the dispersing agent to accomplish this purpose, if desired. Water from a source other than tank 28 may be added to mixer 27 along with, or in place of, that supplied from tank 28 as illustrated at 45. In other words, the water fed to mixer 27 may be

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from any other source, and all, a part, or none may be fed along with the dispersing agent, as desired. It is to be understood, however, that, broadly speaking, advantages from the use of my invention are realized even though the dispersing agent is incorporated in the soap while in "open" or grained condition, with or without subsequent "closing" of the soap, or with or without addition of water.

As illustrated, the mixture from mixer 27 is fed to mixer 32 wherein acidulation of the soap takes place by virtue of feeding thereto mineral acid, conveniently aqueous, e. g., aqueous sulfuric or hydrochloric acid, from tank 33 by means of pump 34 which may be a proportioning pump. Any other means for feeding mineral acid to mixer 32 may be substituted.

In the mixer 32 the mineral acid functions to displace the organic acids from their salts, it being understood that soap is comprised of one or more salts of one or more higher fatty acids, as is well known, and the action of the mineral acid is greatly accelerated by the use of a dispersing agent in accordance with my invention. The fatty acids in the soap are rapidly released, and occur in the mixture as such. Since the higher fatty acids are insoluble in the aqueous phase, a second phase containing the same is formed, and as a result of my invention, formation of separate and distinct phases is greatly facilitated. Moreover, and particularly when the original feed stock is soapstock or gums, the formation of a third phase containing substances other than fatty acids, and which except for their separation would interfere with the subsequent purification of the fatty acids, is outstandingly facilitated. By the use of my invention, a sharper division of phases into an aqueous phase, a fatty acid phase, and a third phase when present, is obtained.

Whereas, without the use of my invention a long time factor running into many hours is involved to effect acidulation, and to cause the formation of phases sufficiently distinct for satisfactory separation, either by gravity or by centrifuging, by the use of my invention this time factor in all cases may be reduced to less than one hour, such as to less than thirty minutes, and typically to ten minutes, making it possible to effect the treatment by continuous flow of the reaction mass through a mixer of the required size in relation to the rate of flow, to provide the required residence time therein. Moreover, this residence or holdup time may be provided in any other manner after intimate mixing of the reaction mass, such as by the use of a holdup tank connected to the outlet of mixer 32 for the flow therethrough of the reaction mass.

While I have illustrated two mixers, namely, mixers 27 and 32 in sequence, it will be understood that any other arrangement or device or devices may be substituted. For example, mixers 27 and 32 might be replaced by a single mixer, particularly if of sufficient length, and while I prefer to add the dispersing agent prior to the addition of the mineral acid, this order of addition may be reversed, or the respective additions may be simultaneous, with the realization of advantages of my invention.

As illustrated, the mixture from mixer 32 is fed to centrifuge 35 wherein separation of phases takes place. If a holding tank is employed, downstream from mixer 32, obviously the feed to centrifuge 35 would be from the outlet of such tank. Since my invention is being described more particularly, and for purposes of illustration, in connection with the production of purified fatty acids from soapstock, I have indicated on the flow sheet of the drawing the separation in the centrifuge 35 of the fatty acid phase on the one hand, from the aqueous phase and the sterol phase (if present) on the other, the fatty acid phase being shown as flowing to distillation tower 36 through line 37, and the aqueous phase and the sterol phase (if present) being shown as flowing from the centrifuge 35 through line 38.

As illustrated, the fatty acid phase is subjected to dis-

tillation in tower 36, whereby the fatty acids are fractionated, the fatty acid fraction leaving tower 36 as illustrated at 41, and the residue leaving tower 36 as illustrated at 42. Any other means for removing fatty acids from the fatty acid phase may be substituted, such as solvent extraction, or the fatty acid phase may be otherwise utilized.

Any desired amount of dispersing agent may be added to the soap mass to be acidulated. Thus from 0.01% to 1% by weight on a dry basis based on the soap mass to be acidulated on a dry basis may be employed, or even more if desired for any reason, such as up to 2 or 3% or even 5%. I find a good working range to be between 0.05% and 1% by weight.

The temperature conditions employed for saponification are usually governed by the rate at which it is desired to saponify, and take into consideration that the reaction is somewhat exothermic. Such temperature conditions usually fall between 170° F. and 212° F., and if saponification is conducted under pressure higher temperature conditions may be employed. The same temperature conditions are typical for the relatively quiescent aging step, and for graining or washing operations. Likewise, any desired temperature conditions may be employed during separation of the phases occurring in the various mixtures produced, temperature conditions falling between 170° F. and 212° F. being typical for the separation of soap mixtures, and 140° F. to 210° F. for the separation of acidulated mixtures.

The dispersing agent may be added to the soap mass under any desired temperature conditions, which may be those existing in the soap mass at the time of the introduction therein of the dispersing agent. Incorporation of aqueous dispersing agent is facilitated at elevated temperatures, e. g., between 140° F. and 210° F. Likewise, acidulation may be carried out under any desired temperature conditions, typical of which are temperatures between 140° F. and 210° F., although temperatures between 165° F. and 195° F. are preferred. Higher or lower temperature conditions may be employed for acidulation if desired for any reason, e. g., between 100° F. and 350° F.

An outstanding advantage of my invention is that it makes possible highly satisfactory acidulation and separation of phases at relatively lower temperatures, e. g., substantially below boiling, and within relatively short times. Both high temperatures and long times contribute considerably to the darkening of the color of the recovered fatty acids.

The temperature conditions employed during distillation are, of course, governed principally by the pressure under which distillation is effected which may be atmospheric, sub-atmospheric or super-atmospheric, as desired.

Pressure conditions at the various points in the process are a matter of choice and judgment, and while they are usually substantially atmospheric, except for the pressures produced by pumps and possibly other devices in effecting continuous flow, they may be sub-atmospheric or super-atmospheric, if desired for any reason. For instance, super-atmospheric pressure would be employed during acidulation to avoid substantial boiling when using temperatures sufficiently high to cause boiling at atmospheric pressure. On the other hand, should sub-atmospheric pressure be employed, the temperature of the acidulation mass should not be so high as to cause substantial boiling at the pressure employed.

The following examples are given by way of illustration and not of limitation.

Example 1

Soapstock resulting from the refining of hydraulically expressed cottonseed oil with sodium carbonate was fed at a rate of 1770 pounds per hour into a system such as is shown in the drawings. The soapstock was mixed at a

temperature between 190 and 200° F. with a 50% aqueous solution of sodium hydroxide at a rate representing 180 pounds per hour of dry NaOH. The resulting stream of saponified mixture was fed through an aging tank with 85 minutes holdup to a centrifuge where it was continuously separated by centrifugal force into spent lye, containing 7.9% NaOH, and soap. The separated stream of soap was washed with an aqueous solution of sodium chloride at a temperature between 170 and 180° F. and at a rate representing 30 pounds per hour of dry NaCl. The mixture was continuously fed to a second centrifuge where it was separated into electrolyte, containing 7% sodium chloride, and soap. The stream of soap discharged from the second centrifuge was fed through a mixer where it was diluted with water to produce a smooth soap mass. Into this mixer was introduced an aqueous solution of the sodium salt of the reaction product of naphthalene, formaldehyde and sulfuric acid, a dispersing agent available in the trade under the trademark Tamol, at a rate of 1.9 pounds per hour on a dry basis. This mass was then delivered in a continuous stream into another mixer maintained at a temperature of about 194° F. together with 192 pounds per hour of 66° Bé. H₂SO₄, the holdup of the mixer being about eight minutes. From this mixer the mixture flowed in a continuous stream to a third centrifuge adjusted to discharge the fatty acid phase on one hand, and a mixture of mineral acid phase and sterol phase on the other hand. The fatty acid phase was delivered from the centrifuge at a rate of 580 pounds per hour. A single distillation of the fatty acid phase resulted in a high yield of purified fatty acids having an acid value of 190 and a Gardner color of 2.

Example 2

Soapstock resulting from the refining of expeller expressed cottonseed oil with sodium hydroxide was fed at a rate of 2480 pounds per hour into a system such as is shown in the drawing. The soapstock was mixed at a temperature between 195 and 205° F. with a 50% aqueous solution of sodium hydroxide at a rate representing 144 pounds per hour of dry NaOH. The resulting stream of saponified mixture was fed through an aging tank with 75 minutes holdup to a centrifuge where it was continuously separated by centrifugal force into spent lye, containing 9.2% NaOH, and soap. The stream of separated soap was washed with an aqueous solution of sodium chloride at a temperature between 175 and 185° F. and at a rate representing 30 pounds per hour of dry NaCl. The mixture was fed in a continuous stream to a second centrifuge where it was separated into spent electrolyte, containing 8% sodium chloride, and soap. The stream of soap discharged from the second centrifuge was fed through a mixer where it was diluted with water to produce a smooth soap mass. Into this mixer was introduced an aqueous solution of the sodium salt of the reaction product of naphthalene, formaldehyde and sulfuric acid, a dispersing agent available in the trade under the trade-name Tamol, at a rate of 1.5 pounds per hour on a dry basis. This mass was then delivered in a continuous stream into another mixer maintained at a temperature about 180° F. together with 162 pounds per hour of 66° Bé. H₂SO₄, the holdup of the mixer being about six minutes. From this mixer the mixture flowed in a continuous stream to a third centrifuge adjusted to discharge the fatty acid phase on one hand, and a mixture of mineral acid phase and sterol phase on the other hand. The fatty acid phase was delivered from the centrifuge at a rate of 600 pounds per hour. A single distillation of the fatty acid phase resulted in a high yield of purified fatty acids having an acid value of 190 and a Gardner color of 2.

Example 3

Soapstock resulting from the refining of cottonseed oil with sodium hydroxide was mixed at a rate of 1140 pounds per hour and at a temperature between 190 and

200° F. with a 50% aqueous solution of sodium hydroxide at a rate representing 189 pounds per hour of dry NaOH. The resulting stream of saponified mixture was fed through an aging tank with 95 minutes holdup to a centrifuge where it was continuously separated by centrifugal force into spent lye, containing 14% NaOH, and soap. The stream of separated soap was fed through a mixer where it was diluted with water to produce a smooth soap mass. Into this mixer was introduced an aqueous solution of the sodium salt of the reaction product of naphthalene, formaldehyde and sulfuric acid, a dispersing agent available in the trade under the trademark Tamol, at a rate of 1.4 pounds per hour on a dry basis. This mass was then delivered in a continuous stream into another mixer maintained at a temperature of about 179° F. together with 240 pounds per hour of 66° Bé. H₂SO₄, the holdup of the mixer being about seven minutes. From this mixer the stream flowed to a second centrifuge adjusted to discharge the fatty acid phase on one hand, and a mixture of mineral acid phase and sterol phase on the other hand. The fatty acid phase was delivered from the centrifuge at a rate of 660 pounds per hour. A single distillation of the fatty acid phase resulted in a high yield of purified fatty acids having an acid value of 187 and a Gardner color of 2.

Example 4

Soapstock resulting from the refining of expeller expressed cottonseed oil with sodium hydroxide was fed at a rate of 1720 pounds per hour into a system such as is shown in the drawing. The soapstock was mixed at a temperature between 200 and 205° F. with a 50% aqueous solution of sodium hydroxide at a rate representing 133 pounds per hour of dry NaOH and a 20% aqueous solution of sodium chloride representing 127 pounds per hour of dry NaCl. The resulting stream of saponified mixture was fed through an aging tank with 37 minutes holdup to a centrifuge where it was continuously separated by centrifugal force into soap and a spent lye containing 8.2% NaOH and 7.8% NaCl. The stream of separated soap was washed with an aqueous solution of sodium chloride at a temperature between 180 and 185° F. and at a rate representing 54 pounds per hour of dry NaCl. The mixture was fed in a continuous stream to a second centrifuge where it was separated into spent electrolyte, containing 9.4% sodium chloride, and soap. The stream of soap discharged from the second centrifuge was fed through a mixer where it was diluted with water to produce a smooth soap mass. Into this mixer was introduced an aqueous solution of the sodium salt of the reaction product of naphthalene, formaldehyde and sulfuric acid, a dispersing agent available in the trade under the trademark Tamol, at a rate of 2.1 pounds per hour on a dry basis. This mass was then delivered in a continuous stream into another mixer maintained at a temperature of about 190° F. together with 306 pounds per hour of 66° Bé. H₂SO₄, the holdup of the mixer being about eight minutes. From this mixer the mixture flowed in a continuous stream to a third centrifuge adjusted to discharge the fatty acid phase on one hand, and a mixture of mineral acid phase and sterol phase on the other hand. The fatty acid phase was delivered from the centrifuge at a rate of 430 pounds per hour. A single distillation of the fatty acid phase resulted in a high yield of purified fatty acids having an acid value of 190 and a Gardner color of 2.

Example 5

Solvent extracted soya bean oil containing 0.5% free fatty acid was refined with sodium hydroxide in a conventional manner. 8040 grams of the resultant soapstock was mixed batchwise at a temperature between 200 and 210° F. with 1439 grams of a 48.7% aqueous solution of sodium hydroxide. The resulting saponified mixture was aged for 90 minutes without agitation, and then fed to a centrifuge where it was separated by centrifugal force

into spent lye, containing 9.1% NaOH, and soap. The separated soap was washed with 232 grams of a 23% aqueous solution of sodium chloride at a temperature between 180 and 190° F. The resulting mixture was fed to centrifuge where it was separated into spent electrolyte, containing 5.3% sodium chloride, and soap. The soap thus separated was fed to a container where it was diluted with water to produce a smooth soap mass. Into this container was introduced 9.1 grams of dry sodium salt of the reaction product of naphthalene, formaldehyde and sulfuric acid, a dispersing agent available in the trade under the trademark Tamol. This mass was then agitated at a temperature of about 190° F. together with 510 grams of 66° Bé. H₂SO₄, for a period of several minutes. The resultant mixture was fed to a centrifuge adjusted to discharge the fatty acid phase on one hand, and a mixture of mineral acid phase and sterol phase on the other hand. A single distillation of the fatty acid phase resulted in a high yield of purified fatty acids having an acid value of 187.

Example 6

Soapstock resulting from the refining of peanut oil with sodium carbonate was fed at a rate of 1620 pounds per hour into a system such as is shown in the drawing. The soapstock was mixed at a temperature between 185 and 195° F. with a 50% aqueous solution of sodium hydroxide at a rate representing 171 pounds per hour of dry NaOH. The resulting stream of saponified mixture was fed through an aging tank with 95 minutes holdup to a centrifuge where it was continuously separated by centrifugal force into spent lye, containing 11% NaOH, and soap. The stream of separated soap was washed with an aqueous solution of sodium chloride at a temperature between 165 and 185° F. and at a rate representing 30 pounds per hour of dry NaCl. The mixture was fed in a continuous stream to a second centrifuge where it was separated into spent electrolyte, containing 7% sodium chloride, and soap. The stream of soap discharged from the second centrifuge was fed through a mixer where it was diluted with water to produce a smooth soap mass. Into this mixer was introduced an aqueous solution of the sodium salt of the reaction product of naphthalene, formaldehyde and sulfuric acid, a dispersing agent available in the trade under the trademark Tamol, at a rate of 1.4 pounds per hour on a dry basis. This mass was then delivered in a continuous stream into another mixer maintained at a temperature of about 176° F. together with 150 pounds per hour of 66° Bé. H₂SO₄, the holdup of the mixer being about nine minutes. From this mixer the mixture flowed continuously to a third centrifuge adjusted to discharge the fatty acid phase on one hand, and a mixture of mineral acid phase and sterol phase on the other hand. The fatty acid phase was delivered from the centrifuge at a rate of 360 pounds per hour.

Example 7

7021 grams of soapstock resulting from the refining of solvent extracted degummed soya bean oil with sodium hydroxide was mixed batchwise at a temperature between 200 and 210° F. with 903 grams of a 48.7% aqueous solution of sodium hydroxide. The resulting saponified mixture was aged for 100 minutes without agitation, and then fed to a centrifuge where it was separated by centrifugal force into spent lye, containing 8.1% NaOH, and soap. The separated soap was washed with 823 grams of a 23% aqueous solution of sodium chloride at a temperature between 180 and 190° F. The resulting mixture was separated by centrifugal force into spent electrolyte, containing 5.5% sodium chloride, and soap. The soap thus separated was fed to a container where it was diluted with water to produce a smooth soap mass. Into this container was introduced 9.1 grams of dry sodium salt of the reaction product of naphthalene, form-

aldehyde and sulfuric acid, a dispersing agent available in the trade under the trademark Tamol. This mass was then agitated at a temperature of about 190° F. together with 510 grams of 66° Bé. H₂SO₄ for a period of several minutes. The resulting mixture was fed to a centrifuge adjusted to discharge the fatty acid phase on one hand, and a mixture of mineral acid phase and sterol phase on the other hand. The fatty acid phase thus obtained was of high quality and yield.

Example 8

Soapstock resulting from the refining of cottonseed oil with sodium hydroxide was fed at the rate of 1500 pounds per hour through a mixer where it was diluted with water to produce a smooth mass. Into this mixer was introduced an aqueous solution of the sodium salt of the reaction product of naphthalene, formaldehyde and sulfuric acid, a dispersing agent available in the trade under the trademark Tamol, at a rate of 1.9 pounds per hour on a dry basis. This mass was then delivered in a continuous stream into another mixer maintained at a temperature of about 195° F. together with 240 pounds per hour of 66° Bé. H₂SO₄, the holdup of the mixer being about seven minutes. From this mixer the mixture flowed continuously to a centrifuge adjusted to discharge the fatty acid phase on one hand, and a mixture of mineral acid phase and sterol phase on the other hand. The fatty acid phase was delivered from the centrifuge at a rate of 480 pounds per hour. A single distillation of the fatty acid phase resulted in a good yield of fatty acids having a Gardner color of 5.

Example 9

6500 grams of soapstock resulting from the refining of cottonseed oil with sodium hydroxide was mixed batchwise at a temperature between 200 and 210° F. with 1910 grams of a 48.5% aqueous solution of sodium hydroxide. The resulting saponified mixture was aged for 80 minutes without agitation, and then fed to a centrifuge where it was separated by centrifugal force into spent lye, containing 10.2 NaOH, and soap. 200 grams of the soap thus separated was placed in a container into which was introduced 0.30 grams of dimethyl octynediol with agitation. This mass was then agitated at a temperature of about 190° F. together with 26.3 grams of 66° Bé. H₂SO₄ for a period of several minutes. The resulting mixture was fed to a centrifuge adjusted to discharge the fatty acid phase on one hand, and a mixture of mineral acid phase and sterol phase on the other hand. The fatty acid phase was obtained in good yield.

Substantially the same results were obtained by substituting other dispersing agents in the above example including various lignosulfonate dispersing agents.

Any other feed stock containing glyceride oil and/or fat, irrespective of its source, and any other dispersing agent for the soap, may be substituted in the foregoing examples to obtain advantages from my invention. Thus any of the glyceride oils and fats normally used in the production of soap for household or industrial use may be substituted, although the economic features of my invention are realized to a much greater extent when the soap to be acidulated is derived from a much inferior source of feed stock.

Soapstocks usually contain some gums, irrespective of whether or not a separate degumming operation has been performed in the refining of glyceride oils and fats, although soapstocks usually contain a higher percentage of gums when a prior degumming operation is not performed.

While my new method of acidulation has been described more particularly as applied to a soap mass which has been completely saponified and grained or washed, and to which it is particularly applicable for the economic

production of high quality fatty acids, it is to be understood that my invention is not limited thereto. Thus my invention is useful when employed in connection with the acidulation of soapstocks which have not been subjected to a saponification treatment, or, whether or not subjected to a saponification treatment, which have not been grained or washed. Certain advantages of my invention may be realized irrespective of the history or source of the soap prior to the acidulation.

It is to be understood that apparatus for carrying out my invention has been illustrated in simplified form for convenience in describing my new process, and that the use of flow controllers, valves, flow meters, gauges, additional pumps, etc., will occur to persons skilled in the art upon becoming familiar herewith, particularly when indicated by good engineering practice.

For convenience in the claims the term "glyceride oil" is used to define any of the glyceride oils and fats.

Having particularly described my invention, it is to be understood that this is by way of illustration, and that changes, omissions, additions, substitutions and/or modifications may be made without departing from the spirit thereof. Accordingly, it is intended that the patent shall cover, by suitable expression in the claims, the various features of patentable novelty which reside in the invention.

I claim:

1. In a process for the production of fatty acids wherein a soap-containing mass is subjected to the action of a mineral acid to free fatty acids from the soap, the step of dispersing said soap-containing mass in aqueous media by action of a dispersing agent to accelerate the action thereon of said mineral acid, and to accelerate the formation into phases of the resultant reaction mass.
2. The process of claim 1 in which the soap-containing mass is soapstock derived from the refining of glyceride oil with an alkaline refining agent.
3. The process of claim 2 in which the glyceride oil is a vegetable oil.
4. The process of claim 2 in which the soap is in at least substantially completely saponified condition.
5. The process of claim 3 in which the soap is washed with an electrolyte solution prior to contact with the mineral acid.
6. The process of claim 4 in which the soap is dispersed after the washing but prior to contact with the mineral acid.
7. The process of claim 5 in which the soap is converted into a smooth mass by the addition of water after the washing but prior to contact with the mineral acid.
8. The process of claim 6 in which the mineral acid is sulfuric acid.
9. The process of claim 1 in which the soap in a continuously flowing stream is subjected to the action of the mineral acid to release fatty acids therefrom, and thereafter is subjected in a continuously flowing stream to centrifugation to separate fatty acids.
10. The process of claim 8 in which the fatty acids are separated in a phase apart from other phases present.
11. The process of claim 9 in which the separated fatty acid phase is subjected to distillation to obtain a fatty acid fraction.
12. The process of claim 1 in which the soap is dispersed prior to contact with the mineral acid.
13. The process of claim 1 in which the acid is sulfuric acid.

References Cited in the file of this patent

UNITED STATES PATENTS

1,862,037	Schlenkes	June 7, 1932
2,285,902	Christmann	June 9, 1942
2,319,929	Hoffman et al.	May 25, 1943