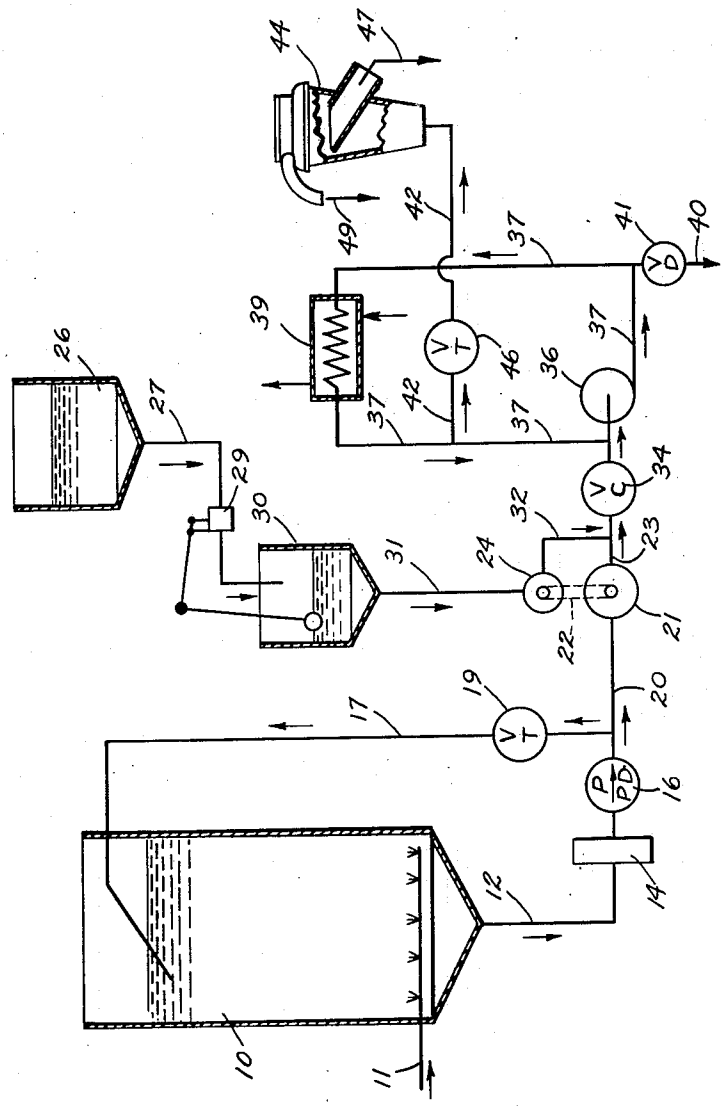
Oct. 23, 1951 J. W. BODMAN ET AL 2,572,102

CENTRIFUGAL SEPARATION OF KETTLE SOAP

Filed Sept. 16, 1947 2 SHEETS—SHEET 1

FIG. I.



INVENTOR.
JOHN W. BODMAN,
FREDERICK J. SQUIRE &
BY GEORGE W. BUSBY JR.

Campbell, Brumbaugh & Free
THEIR ATTORNEYS.

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FIG. 3.

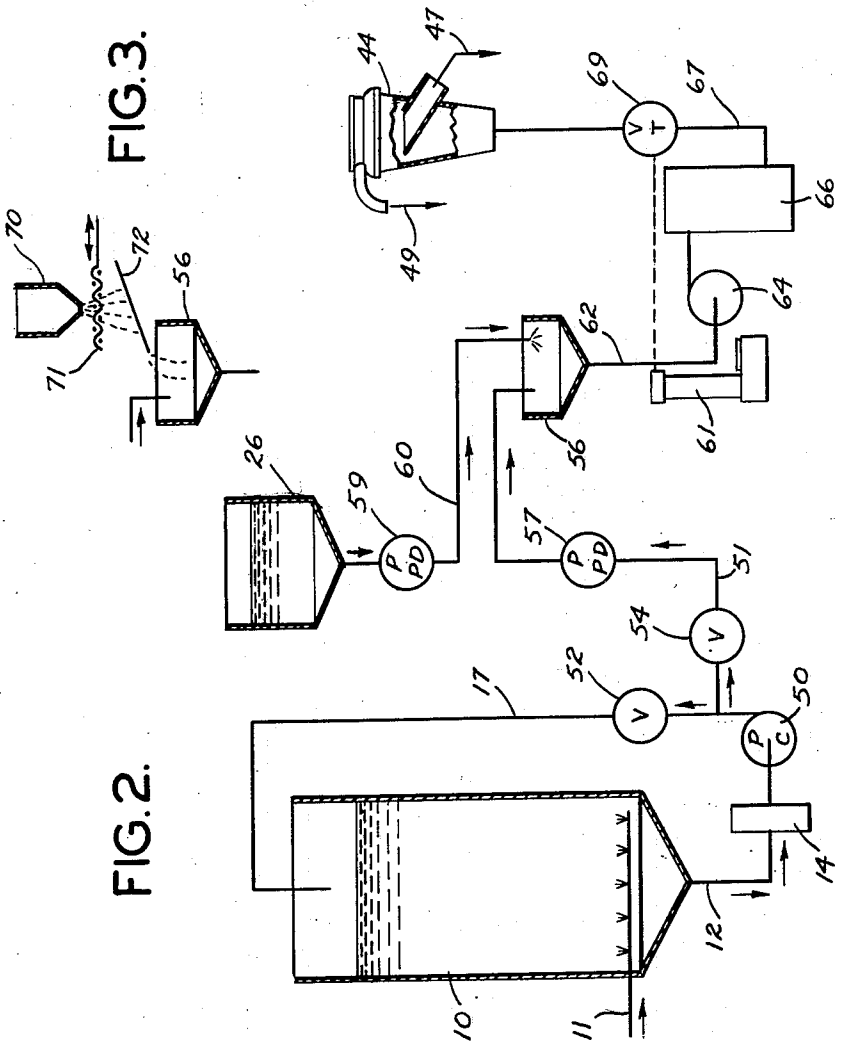

FIG. 2.

INVENTOR.
JOHN W. BODMAN,
FREDERICK J. SQUIRE &
BY GEORGE W. BUSBY, JR.

Campbell, Brumbaugh & Free

THEIR ATTORNEYS.

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CENTRIFUGAL SEPARATION OF KETTLE SOAP

John W. Bodman, Winchester, and Frederick J. Squire, Milton, Mass., and George W. Busby, Jr., Baltimore, Md., assignors to Lever Brothers Company, Cambridge, Mass., a corporation of Maine

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8 Claims. (Cl. 260—418)

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The present invention relates to the manufacture of soap and more particularly to the rapid separation of a batch of kettle soap into neat soap and a nigre or pitch lye.

The conventional method of making soap is to boil fats and oils, referred to in the soap making art as glycerides, in large kettles with the requisite amount of caustic soda. This step is usually referred to as the saponification or the killing change inasmuch as most of the glycerides are saponified in this step. A number of further so-called changes are subsequently carried out, during which the saponified materials are washed by means of salt solutions, strong caustic solutions and weak caustic solutions in order to insure a complete saponification of the glycerides and to remove glycerine and excess electrolyte from the soap. These changes have the general purpose of progressively assuring complete saponification of the glycerides and the removal of undesirable materials from the saponified soap product.

In the course of each change, the mass is boiled by blowing open steam through coils at the bottom of a kettle. This treatment causes the soap mass to swell and brings about intimate mixing of the soap and lye solution. At the end of each change, the steam is shut off and the mass is allowed to settle. A grained soap curd rises to the top and an aqueous phase or lye forms in the bottom of the kettle from whence it is withdrawn.

The finishing operation in the conventional soap making procedure consists in adding a certain amount of water and, if desired, small quantities of lye or salt, to grained soap curd produced by the preceding operations, boiling the mixture of soap curd and water with live steam in a kettle and then allowing the mass to settle so that a neat soap of the desired composition rises to the top and a nigre separates by gravity to the bottom of the kettle. A nigre usually contains an appreciable amount of fatty acids in the form of soap, as well as excess electrolyte, coloring matter, and dirt.

Although the nigre begins to separate by gravity immediately, the time required to settle the nigre completely usually varies between about forty and sixty hours. As a consequence, the kettle in which the operation of settling is carried out is unavailable for other uses. Furthermore, the pipe through which the neat soap is taken off at the end of the settling period must necessarily be kept well above the nigre line so as not to contaminate the neat soap withdrawn with any of the nigre in the bottom of the kettle. The result of this necessary practice is that a considerable amount of neat soap is left with the nigre and must be reprocessed.

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Some attempts have been made in the past to accelerate this settling process by employing a centrifuge. Difficulties have, however, been encountered in such attempts because as soon as the steam is turned off in the finishing operation in the kettle, a nigre immediately begins to separate by gravity, and it has not been found possible to maintain the mass in a homogeneous condition by means of ordinary agitation, such as the use of mechanically driven agitators or pumps with circulating lines. Furthermore, and perhaps due in part at least to the aforementioned difficulty, it was found difficult to maintain a constant rate of feed to the centrifuge. The conditions of homogeneity and constant rate of feed are essential to the successful operation of a centrifuge.

It has now been found that it is possible to condition a batch of kettle soap from one of the changes prior to the finishing operation, and then to treat the conditioned soap so that a nigre or lye can be separated rapidly from neat soap. The conditioning step, which is carried out at or near the boiling temperature of the soap, comprises adding water and, if desired, small quantities of electrolyte, to produce a soap mass having a total fatty acid content of between about 40 and 66%, preferably between about 50 and 59%, by weight of the composition, the electrolyte content being sufficient to maintain the conditioned soap in a homogeneous condition by means of a pump and circulating lines or other mechanical means of agitation. The subsequent step of treating the conditioned soap is carried out by adding to, and mixing with the conditioned soap measured amounts of electrolyte sufficient to further reduce the total fatty acid content of the mixture to between about 38 and 62%, preferably between about 45 and 55%, by weight and then charging it to a centrifuge at a uniform rate of feed. The soap-electrolyte mixture is maintained at a temperature above about 180° F. because at lower temperatures its viscosity would become too great for separation with reasonable efficiency. Optimum efficiency of separation is attained by maintaining the mixture at a temperature between about 190° and 200° F. The electrolyte may be added in the form of a solution or in dry form and its admixture with the conditioned soap converts the latter into a somewhat less homogeneous mixture of neat soap

and nigre or lye that is susceptible to efficient separation by the application of centrifugal force, the neat soap and nigre or lye being separated on the light and heavy sides, respectively, of the bowl. By this means a kettle of soap may be finished and settled in a battery of centrifuges at a rate of approximately 2500 pounds of neat soap (63% total fatty acids) per hour per centrifuge, whereas the normal kettle settling process would usually require from 50 to 60 hours per kettle.

It has also been found that by maintaining the bowl adjustment at the centrifuge constant and changing the electrolyte content of the soap mixture, the proportions of soap removed from the mass in the heavy effluent may be varied from only a trace of fatty acids to amounts up to and sometimes exceeding 35% of the total fatty acids in the soap mass. Thus, the ratio electrolyte to soap in the soap-electrolyte mass will determine the constitution of the heavy effluent. When the proportion of electrolyte added is low, a nigre consisting of water, salt, caustic soda, glycerol and soap-containing coloring materials is obtained. When the proportion of electrolyte added is high, the soap that is separated tends to become grained, and the heavy effluent will be a pitch water or lye containing water, salt, caustic soda, glycerol and relatively small amounts of soap. The heavy effluent is designated as a nigre or a pitch, depending on the amount of soap it contains.

The process of this invention is peculiarly adapted to the production of a uniform neat soap. Once a set of conditions, namely, the total fatty acid content and electrolyte content of a conditioned soap, and the amount of electrolyte with which it is subsequently treated, has been found to yield a finished soap of a desired constitution, the same conditions can be reproduced in the conditioning and subsequent treatment of other batches of kettle soap, even though they may vary considerably, to produce a uniform grade of finished soap. Since the first two conditions can be determined analytically and the third is quantitative, the personal factor in the final finishing of the soap is effectively eliminated.

These and further advantages of the invention will become apparent from the following detailed description and in the light of the attached drawing, in which:

Figure 1 is a flow sheet illustarting a preferred method of carrying out the process of the invention;

Figure 2 is a flow sheet illustrating another method of carrying out the process of the invention; and

Figure 3 is a schematic view of a portion of the equipment illustrated in Figure 2 whereby the process can be carried out with the addition of dry electrolyte.

Referring now to Figure 1, a batch of kettle soap from a change prior to the finishing operation is introduced into a soap relay tank 10 in any suitable manner and conditioned by adding a measured quantity of water and, if desired, small amounts of electrolyte, such as salt or caustic. The soap mass so conditioned is boiled with live steam entering tank 10 by way of line 11 and is closed thereby so that neither a pitch lye nor a nigre will settle out while the mass is agitated by pump circulation or the like. The conditioned soap mass is withdrawn from the bottom of tank 10 through line 12, passed through a strainer 14 and pumped by means of a positive displacement pump 16 through an insulated circulating line 17 and throttle valve 19 to be reintroduced into the top of tank 10.

The conditioned and circulating soap mass is bled off circulating line 17 by way of a line 20 that is preferably, but not necessarily, of smaller cross section than circulating line 17. The soap mass passing through line 20 enters a positive displacement pump or other metering device 21, connected by means of a chain and sprocket or other suitable means indicated generally at 22, to a positive displacement pump 24 which pumps a predetermined proportion of electrolyte from a tank 26 through line 27, valve 29, constant level tank 30 and line 31 into line 23 by way of line 32.

The conditioned soap and the electrolyte pass from positive displacement pumps 21 and 24 through a check valve 34, to a centrifugal pump 36 in a second circulating line 37, including a heat exchanger 39, and a drain 40 provided with a drain valve 41. The conditioned soap and electrolyte are thoroughly mixed in circulating line 37, maintained in circulating motion by means of centrifugal pump 36 and heated by passage through heat exchanger 39 to maintain it at a temperature of not less than about 180° F. and preferably at between 190° and 200° F.

The conditioned soap-electrolyte mixture circulating in circulating line 37 is drawn off at a uniform rate by way of line 42 and introduced into one or more centrifuges 44 after passing through a throttle valve 46. Neat soap is removed from the light side of the bowl of centrifuge 44, as shown diagrammaticaly by arrow 47, and a nigre or lye is removed from the heavy side, as shown diagrammatically by arrow 49.

The embodiment illustrated in Figure 2 likewise includes the introduction of a batch of kettle soap from a change prior to the finishing operation into a soap relay tank 10 and the conditioning of said soap by the addition of a measured quantity of water, and if desired, small amounts of electrolyte such as salt or caustic. The soap mass in tank 10 so conditioned is boiled with live steam entering tank 10 by way of line 11 and is closed thereby so that neither a pitch lye nor a nigre will settle out while the mass is agitated by pump circulation or the like. The conditioned soap mass is withdrawn from the bottom of the tank 10 through line 12, passed through a strainer 14 and pumped by means of a centrifugal pump 50 through an insulated circulating line 17 into the top of tank 10. The conditioned and circulating soap mass is bled off circulating line 17 into line 51 at a predetermined rate and is introduced into a feed and mixing funnel 56 by means of a positive displacement pump 57. Valves 52 and 54 may be provided in lines 17 and 51, respectively, to insure a satisfactory rate of circulation through line 17 and a constant head on the suction side of pump 57. At the same time a predetermined proportion of electrolyte from tank 26 is introduced by way of a second positive displacement pump 59 and line 60 into the mixing and feed funnel 56, the desired proportion of conditioned soap and electrolyte entering funnel 56 being maintained by proper adjustment of the speeds of positive displacement pumps 57 and 59.

Funnel 56 is supported by a weight controlling device shown schematically at 61. The soap-electrolyte mixture from funnel 56 passes through line 62 by way of a centrifugal pump 64 to a mixer and heat exchanger 66 wherein the condi-

tioned soap and electrolyte are thoroughly mixed and maintained at a temperature of not less than about 180° F. and preferably between about 190° and 200° F. The conditioned soap-electrolyte mixture thereupon passes through line **67** at a rate determined by throttle valve **69**, which is air actuated and controlled through the medium of a needle valve on the lever arm of weight controlling device **61** to maintain a constant level in funnel **56**, and passed into a centrifuge **44** at a uniform rate of feed. Neat soap is removed from the light side of the bowl of centrifuge **44** as shown schematically by arrow **47** and a nigre or lye is removed from the heavy side as shown schematically by arrow **49**.

If it is desired to add the electrolyte in the dry state rather than in the form of a solution, a source of dry electrolyte **70**, shown schematically in Figure 3, is provided to deposit the electrolyte upon a screen **71** which can be vibrated or shaken in any suitable manner and at a predetermined rate to deposit the desired proportion of dry electrolyte upon a plate **72** from whence it will be guided into funnel **56**.

It is to be understood that many changes and modifications can be made in the apparatus specifically described and in the manner in which the various steps are carried out, without departing from the scope of this invention. Thus, for example, the proportioning systems described with reference to the figures of drawing may be interchanged or substituted by any other suitable proportioning means; a by-pass line may be provided for the conditioned soap mass from line **20** directly to the check valve **34**; any mechanical, multistage, or orifice type of mixer may be substituted for circulating line **37** and centrifugal pump **36** of Figure 1 or the mixer and heat exchanger **66** of Figure 2; and the electrolyte added to the soap mass in tank **10** need not be the same as that added to the conditioned soap mass by means of the metering arrangement.

Furthermore, it is contemplated that the operations described herein may, if desired, be carried out a number of times. Thus, for example, the process could first be carried out so as to separate a grained soap and pitch water or lye, and then the operation may be repeated so as to separate neat soap and a nigre.

The foregoing changes and modifications are illustrative of but a few of the many that will readily become apparent to those skilled in the soap making art upon reading this description.

Without intending to limit the scope of the invention in any way, the following examples are included to more specifically illustrate the procedures of and the results obtainable by means of the present invention.

Example 1

A batch of 8000 lbs. of kettle soap was boiled in a soap relay tank, conditioned by adding 180 lbs. of water, and maintained in a homogeneous state by circulating it through an insulated circulating line by means of a positive displacement pump. The conditioned soap mass was found, upon analysis, to have a total fatty acid content of 58.75% and to contain 0.42% free Na_2O, 0.23% Cl and 0.28% glycerine. 2910 lbs. per hour of the circulating soap mass were then bled off the circulating line and mixed with 165 lbs. per hour of a 20% aqueous salt solution. The conditioned soap and added salt solution were then continuously and thoroughly mixed in a circulating line by means of a centrifugal pump and maintained at a temperature of between 190° and 200° F. by the addition of heat in a heat exchanger. This circulating mixture was bled off at a uniform rate and discharged into a centrifuge at a rate of 3075 lbs. per hour.

It was found that 2688 lbs. per hour of neat soap were separated on the light side of the bowl of the centrifuge and 387 lbs. per hour of pitch lye were separated on the heavy side of the centrifuge. The neat soap was found to have a total fatty acid content of 63.95% and to contain 0.15% free Na_2O, 0.28% Cl and 0.26% glycerine. The pitch lye was found to contain 3.04% sodium hydroxide and 7.8% sodium chloride.

Example 2

A bath of 8000 lbs. of kettle soap was boiled in a soap relay tank, conditioned by adding 182 lbs. of water, 12 lbs. of sodium chloride and 6 lbs. of a 50% sodium hydroxide solution and maintained in a homogeneous state by circulating it through an insulated circulating line by means of a centrifugal pump. The conditioned soap mass was found, upon analysis, to have a total fatty acid content of 50.99%, and to contain .08% free Na_2O, 1.42% Cl and 0.83% glycerine. 2186 lbs. per hour of the circulating soap mass were then continuously bled off and passed into a feed funnel mounted on a weight controlling device. An 11.2% aqueous salt solution was added at a rate of 294 lbs. per hour to the conditioned soap mass in the feed funnel. The conditioned soap and added salt solution were then thoroughly mixed and maintained at a temperature of 190° to 200° F. in a mixer and heat exchanger. The mixture was discharged to a centrifuge at a uniform rate of 2480 lbs. per hour.

It was found that 1845 lbs. per hour of the neat soap were separated from the light side of the centrifuge and 635 lbs. per hour of nigre were separated from the heavy side. The neat soap was found to contain 62.28% total fatty acids, .02% of free Na_2O, 0.65% Cl and 0.49% glycerine. The nigre was found to contain 5.21% total fatty material, 0.32% sodium hydroxide and 11.75% sodium chloride.

We claim:

1. In the manufacture of soap, a process which comprises conditioning a batch of substantially completely saponified kettle soap by adding thereto sufficient water to reduce the total fatty acid content to from about 50 to about 59% by weight, agitating said conditioned soap mass to maintain it in a homogeneous condition, withdrawing a continuous stream of said conditioned soap from said mass, adding to said stream of conditioned soap a continuous stream of electrolyte proportioned to said stream of soap to further reduce the total fatty acid content to from about 45 to about 55% by weight, intimately mixing and agitating said soap and said electrolyte, maintaining said mixture at a temperature of about 190° to 200° F., and centrifuging said soap-electrolyte mixture to separate neat soap therefrom.

2. In the manufacture of soap, a process which comprises conditioning a batch of substantially completely saponified kettle soap by adding thereto sufficient water to reduce the total fatty acid content to from about 50 to about 59% by weight, agitating said conditioned soap mass to maintain it in a homogeneous condition, withdrawing a continuous stream of said conditioned soap from said mass, adding to said stream of conditioned soap a continuous stream of electrolyte propor-

tioned to said stream of soap to further reduce the total fatty acid content to from about 45 to about 55% by weight, intimately mixing and agitating said soap and said electrolyte, maintaining said mixture at a temperature above about 180° F., and centrifuging said soap-electrolyte mixture to separate neat soap therefrom.

3. In the manufacture of soap, a process which comprises conditioning a batch of substantially completely saponified kettle soap by adding thereto sufficient water and electrolyte to reduce the total fatty acid content to from about 50 to about 59% by weight, agitating said conditioned soap mass to maintain it in a homogeneous condition, withdrawing a continuous stream of said conditioned soap from said mass, adding to said stream of conditioned soap a continuous stream of electrolyte proportioned to said stream of soap to further reduce the total fatty acid content to from about 45 to about 55% by weight, intimately mixing and agitating said soap and said electrolyte, maintaining said mixture at a temperature of about 190° to 200° F., and centrifuging said soap-electrolyte mixture to separate neat soap therefrom.

4. In the manufacture of soap, a process which comprises conditioning a batch of substantially completely saponified kettle soap by adding thereto sufficient water and electrolyte to reduce the total fatty acid content to from about 50 to about 59% by weight, agitating said conditioned soap mass to maintain it in a homogeneous condition, withdrawing a continuous stream of said conditioned soap from said mass, adding to said stream of conditioned soap a continuous stream of electrolyte proportioned to said stream of soap to further reduce the total fatty acid content to from about 45 to about 55% by weight, intimately mixing and agitating said soap and said electrolyte, maintaining said mixture at a temperature above about 180° F., and centrifuging said soap-electrolyte mixture to separate neat soap therefrom.

5. In the manufacture of soap, a process which comprises conditioning a batch of substantially completely saponified kettle soap by reducing the total fatty acid content thereof to from about 50 to about 59% by weight, agitating said conditioned soap mass to maintain it in a homogeneous condition, withdrawing a continuous stream of said conditioned soap from said mass, adding to said stream of conditioned soap a continuous stream of aqueous electrolyte proportioned to said stream of soap to further reduce the total fatty acid content to from about 45 to about 55% by weight, intimately mixing and agitating said soap and said electrolyte, maintaining said mixture at a temperature above about 180° F., and centrifuging said soap-electrolyte mixture to separate neat soap therefrom.

6. In the manufacture of soap, a process which comprises conditioning a batch of substantially completely saponified kettle soap by reducing the total fatty acid content thereof to from about 50 to about 59% by weight, agitating said conditioned soap mass to maintain it in a homogeneous condition, withdrawing a continuous stream of said conditioned soap from said mass, adding to said stream of conditioned soap a continuous stream of dry electrolyte proportioned to said stream of soap to further reduce the total fatty acid content to from about 45 to about 55% by weight, intimately mixing and agitating said soap and said electrolyte, maintaining said mixture at a temperature above about 180° F., and centrifuging said soap-electrolyte mixture to separate neat soap therefrom.

7. In the manufacture of soap, a process which comprises conditioning a batch of substantially completely saponified kettle soap by reducing the total fatty acid content thereof to from about 40 to about 66% by weight, agitating said conditioned soap mass to maintain it in a homogeneous condition, withdrawing a continuous stream of said conditioned soap from said mass, adding to said stream of conditioned soap a continuous stream of electrolyte proportioned to said stream of soap to further reduce the total fatty acid content to from about 38 to about 62% by weight, intimately mixing and agitating said soap and said electrolyte, maintaining said mixture at a temperature above about 180° F., and centrifuging said soap-electrolyte mixture to separate neat soap therefrom.

8. In the manufacture of soap, a process which comprises conditioning a batch of substantially completely saponified kettle soap by reducing the total fatty acid content thereof to from about 50 to about 59% by weight, agitating said conditioned soap mass to maintain it in a homogeneous condition, withdrawing a continuous stream of said conditioned soap from said mass, adding to said stream of conditioned soap a continuous stream of electrolyte proportioned to said stream of soap to further reduce the total fatty acid content to from about 45 to about 55% by weight, intimately mixing and agitating said soap and said electrolyte, maintaining said mixture at a temperature above about 180° F., and centrifuging said soap-electrolyte mixture to separate neat soap therefrom.

JOHN W. BODMAN.
FREDERICK J. SQUIRE.
GEORGE W. BUSBY, JR.

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