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METHOD FOR THE CONTINUOUS PURIFICATION OF SOAP

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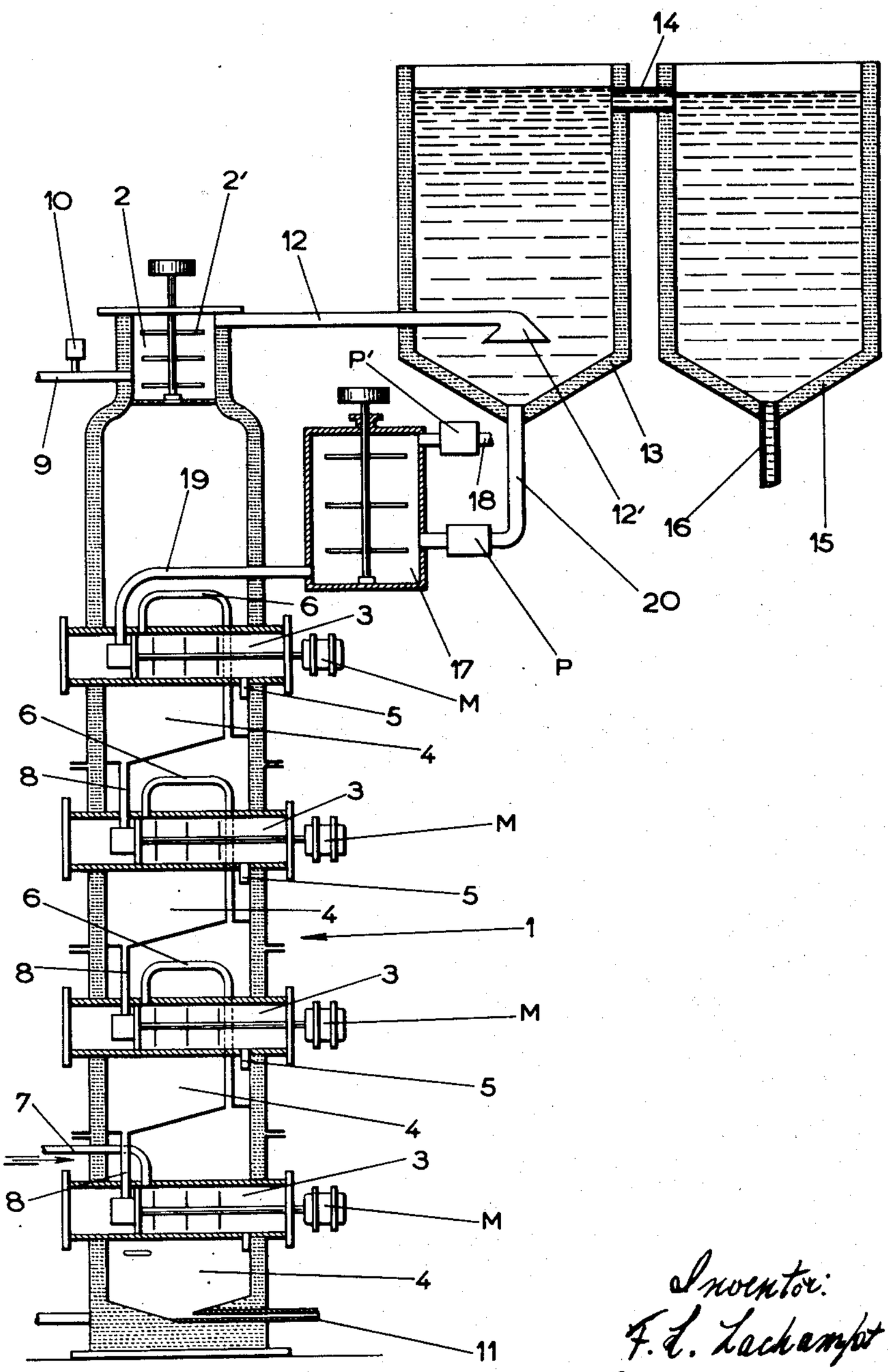

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METHOD FOR THE CONTINUOUS PURIFICATION OF SOAP

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This invention is concerned with improvements in the methods of continuous purification of soap, and with an apparatus for use in applying said improvements.

An object of my invention is to provide a method of continuously and automatically purifying the reaction products obtained when a mixture of fatty substances and/or acids or the like is mixed with a caustic alkali such as caustic soda.

Another object of my present invention is to provide a method of continuously purifying a soap obtained in a concentrated anisotrope phase, such as neat soap or kettle wax.

A further object of my invention is to provide an arrangement for applying my improved method.

A number of methods have been designed already to purify continuously a soap mixture. Thus, in the U. S. patent to Scott, No. 2,300,749, filed May 6, 1940, a method has been described wherein purification is achieved simultaneously with saponification. Also, in my copending patent applications 672,082 and 672,083 both filed May 24, 1946, a method has been described wherein a soap, after having been saponified completely, is purified by washing with a solution of an alkaline chloride; thereafter washing is effected by a solution of an alkali for eliminating the chloride in excess, and then the purificatiton is completed by neutralising the mixture with an acid, according to any convenient means adapted to lower the alkalinity of a commercial soap to a permissible value.

It is well-known, in the conventional method of kettle production, that after the soap has been washed by a brine and the used brine has been removed, the last purifying step consists of adding water in a convenient amount. This is the conventional "fitting" or "finishing" step, wherein the soap separates into two layers or phases; an upper layer consisting of a "neat soap" which is transformed thereafter into a commercial product, and a lower layer consisting of the "nigre," that is a solution of soap in an alkaline water brine.

Kettle fitting is a discontinuous and slow operation; about forty-eight hours are required for the decantation to take place. Such a process, therefore, is in no way suitable in conjunction with a process of continuous boiling of soap, since

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the length of time required for the decantation would counterbalance completely the advantages resulting from the continuity of the preceding operations. That is the reason why many attempts have been made to design methods and processes of continuous fitting or finishing of soap usable with a method of continuous preparation of soap. As far as I am aware, however, none of these methods has been successful.

I have now designed a novel and improved method of continuous finishing of soap, derived from the conventional method used in the kettle soap preparation, and have determined accurately the various parameters which act to change the conditions of the fitting or finishing operation.

According to my invention, the soap is continuously washed in a tower comprising a series of separated stages and the nigre solution drawn off the last washing stage is treated with a brine causing said nigre solution to separate into a poorer brine and into neat soap, both of which are recycled.

Preferably the soap is obtained previously by reacting a mixture of fatty substances, of fatty substances and acids, or fatty substances and resinic acids, or else fatty or resinic acids and soapstock, with a caustic alkali, particularly caustic soda the concentration of which is preferably higher than 30%. Such a reaction gives a completely saponified soap, that is a soap in which no free fatty substance remains present.

More generally, the invention has for one object to purify a soap obtained in a concentrated anisotrope phase, such as neat soap or kettle wax. In practice, the concentration of such a soap is comprised within 65% to 75%; in some particular cases, however, for instance in the case of sodium stearate, the diagram of which was given by McBain and Lee, the concentration may be as low as about 48%.

According to my invention, the soap is continuously and methodically washed by a salt brine having a concentration slightly higher than the limit concentration corresponding to the composition of the purified soap under consideration, in a tower comprising a series of stages each provided with both a mixing and a decanting zone; then, the soap is continuously "finished" by continuous decantation of neat soap at the top of a

kettle, while the mixture in the lower part of said kettle is drawn off the bottom thereof, means being provided for controlling the rate of flow of the drawn off mixture. The quantity of the portion thus drawn off is chosen to comprise some of the neat soap from the upper part of the kettle with the whole nigre deposited at the lower part thereof and this portion is treated with a brine having a concentration higher than that of the first-mentioned washing solution, said concentration being so determined, that said portion be separated completely into neat soap and a poorer brine, the concentration of which precisely equals that of said first-mentioned brine; the neat soap and the brine so obtained are recycled separately in the washing stage which immediately precedes the finishing stage, whereby the neat soap is added to that previously formed and separated, while the poorer brine obtained may be used for washing the soap.

Other details of the method according to my present invention will be described now, which influence the good operation thereof.

The temperature of all the fluid solutions present in the cycle must be maintained within the range 80° to 90° C., for instance 85° C. It is desirable indeed that no vaporisation of the water takes place, and thus the temperature must be kept far below 100° C. taking in account the fact that the structural transformation of the soap during the water addition is exothermic.

Obviously the concentration of the chloride solution used for the washing must be slightly greater than that of the limit brine corresponding to the purified soap, and that concentration must be chosen in such a way that the chloride solution only dissolves the oxyacidic soap, which may cause growing, and the low molecular weight soaps, the existence of which is undesirable. In the present specification, a "limit brine with respect to a soap" is defined as a brine having such a concentration that said soap is not dissolved thereby, although said soap could be dissolved by a brine having a lesser concentration.

The ratio of the volume of the washing solution used to the volume of the fatty acids present in the soap to be washed must be 30 to 50 parts washing solution to every 70 parts fatty acids, and generally said ratio equals 40/70.

The washing is effected by methodical counterflow of the soap and the brine with respect to each other, and must be carried out in a number of succeeding stages, preferably four; in each stage the soap and the brine are intimately mixed, then the mixture is directed to a decantation zone, wherein 80% to 90% of the washing solution separates to be recycled in the immediately preceding stage for use in that stage.

When leaving the last washing stage, therefore, the soap contains substantially 10% of brine and for the finishing operation, 10 to 20 parts water must be added to 100 parts of soap, that is an average amount of 15%.

The occluded gases are removed from the washing brine and from the finishing water, respectively, before the use thereof, in order to avoid bubbling out and foaming which would be detrimental both to the washing and the finishing operations.

Preliminarily a small quantity of a caustic alkali is added to the finishing water, preferably in an amount comprised between 0.2% and 2%. As a matter of fact it has been found that the free alkali content of the liquid soap is a function of the free alkali content of the finishing water.

For instance, the following table gives the values measured on a commercial soap and indicates how the alkalinity is distributed between the finishing water, the soap and the nigre present in the solution:

TABLE

Percent alkalinity

finishing water	soap	nigre
.5	.005	.02
1	.005	.11
1.5	.02	.22
2	.04	.33

Therefore, the addition of some alkali to the finishing water permits the obtention of a soap having a perdetermined free alkali content; said addition further prevents a carbonic foaming due to the possible action of a carbonated alkali contained in the soap on the fatty acids, said action being caused to manifest itself by the fact that the soap becomes hydrolysed by a neutral brine; the resultant washing with an alkaline water is completed to such a point that, starting from a very alkaline soap, a completely neutral soap, or even a partially acid soap may be obtained.

The washed soap and the finishing water are mixed during a time amounting to a maximum of ten minutes, generally two minutes.

The soap is introduced in the kettle at a level below the middle-height thereof, for instance at a fifth of the kettle height, as measured from the bottom of the kettle, through a flared-out opening, in such a way that the flowing rate of the soap be less than one centimeter per second, in order to prevent the formation of any eddy current detrimental to the decantation.

The volume of solution drawn off the decantation kettle is greater than the volume of nigre theoretically formed in said kettle, by about 20%. Thus the drawing off of the whole nigre present in the kettle is secured, said nigre being mixed with some neat soap, which has no importance since the total volume drawn off is recycled thereafter into the last washing stage.

The drawn off liquid must undergo a change of structure and be separated into neat soap and poorer brine before it is recycled in the mixer of the last washing stage; this is obtained by increasing the salt content of the mixture. Preferably the salt content is increased by adding fresh brine having such a concentration that the neat soap thus separated from the mixture be in equilibrium with the poorer brine thus obtained, the concentration of which is very near the limit concentration below which the soap may be dissolved thereby.

If, for instance, the limit concentration for the brine corresponding to a given soap is 13.5%, the nigre will be separated by means of such a brine that, after separation, the nigre mixture presents a layer of neat soap and a layer of brine having a concentration of 13.5%. Experiments have shown that, in such a case, one part of the nigre is to be treated with one part of a brine having a 15% concentration. More generally, I have found that the concentration of the brine used for the separation of the nigre mixture must be higher than the concentration of the limit brine by 1% to 5%.

An arrangement for use in conjunction with the method according to my invention comprises

in combination: a series of intercommunicating washing stages, each of said stages including a mixer, a settling compartment, a pipe between said mixer and said settling compartment and pipes for circulating the soap and the brine in counterflow with respect to each other from said settling compartments respectively in the mixer of the next higher stage and in the mixer of the next lower stage; means for admitting soap into the mixer of one end stage of said series; means for admitting washing brine into the mixer of the opposite end stage in said series; a chamber provided with mixer means and pipes for admitting water and flowing soap out the settling compartment of said opposite end stage respectively; a finishing kettle; a tube connected to said mixer means and opening in said finishing kettle at a level below the middle-height thereof through downwardly directed, outwardly flared end portion; an upper pipe for removing the neat soap from said finishing kettle; a lower pipe for removing the mixture in the lower part of said finishing kettle; a variable flow volumetric pump inserted in said lower pipe; a mixer tank fed from said lower pipe; a pipe for introducing a brine in said mixer tank, said pipe being connectable successively to two sources of supply of brines having different concentrations respectively; a second variable flow volumetric pump inserted in said last-mentioned pipe; a third pipe connecting said mixer tank with the mixer in the last washing stage immediately preceding said chamber with mixer means; means for removing preliminarily the occluded gases from the liquids present, and means for maintaining the temperature of the fluids and apparatus within the range 80 to 90° C.

The arrangement of such a device will be best understood from the following description of an illustrative embodiment theerof, taken in connection with the annexed sheet of drawing, wherein the single figure schematically shows such an arrangement in vertical cross-section.

The described purifying arrangement illustrated in the figure is a part of a plant for the continuous preparation of soap, which has not been shown for the sake of clarity. The arrangement comprises a four-stage washing tower 1 including a chamber 2 provided with mixing or stirring means 2' at the upper end of said tower.

Each washing stage in said tower comprises a mixer compartment 3 and a decantation compartment 4 connected together through a pipe 5. The mixers 3 each are driven from respective electrical motors M. A pipe 6 connects the mixer compartment in every stage, except the uppermost one, with the mixer 3 in the next preceding stage. Another pipe 6 connects the decantation compartment 4 in each stage with the mixer 3 in the next following stage, except for the lowermost stage. The soap to be washed is caused to flow in the lowermost stage through a pipe 7, and the water for "finishing" or "fitting" the soap is supplied in the uppermost stage in the tower 1 through a pipe 9 to which a thermostat 10 is connected, the function of which will be discussed hereafter.

When the soap has been washed, the used brine is eliminated to waste through the tube 11. The liquid soap flows off the chamber 2 through a pipe 12 which terminates in a flared out, downwardly directed mouthpiece 12' within a decanting kettle 13.

Through an upper tube 14, the kettle 13 is connected with a kettle 15 for receiving the purified soap; said soap may be taken off through the lower pipe 16 at the bottom of the kettle 15.

A variable flow volumetric pump P is inserted in a pipe 20 passing through the bottom wall of the kettle 13 at one end and opening in a mixer tank 17 at the other end. The mixture in the kettle 13 may be taken off from said pump P.

The mixer tank 17 is connected to a suitable supply of brine through a pipe 18 provided with a pump P'. The mixture contained in the tank 17 may flow to the mixer 3 in the uppermost washing stage in the tower through a pipe 19.

The purification is carried out as follows:

When starting, the pump P is stopped, whereas the pump P' is operated and urges—through the conduct 18, the mixer 17 and the pipe 19—into the mixer 3 of the highest washing stage a salt solution having a concentration slightly higher than the previously defined limit concentration, for instance a 13.5% chloride solution. Said chloride solution (or brine) further flows from the mixer 3 of said highest stage into the settling compartment 4 of this stage through the pipe 5. This brine thereafter flows through the pipe 8 into the mixer 3 of the next stage which is located below, and then from this mixer into the settling compartment 4 of same stage through the pipe 5; so the brine is flowing from a stage to the next and lower one to finally reach the mixer 3 of the lowest stage wherein it comes into contact with the soap to be purified. The latter is driven back into the washing tower 1 by a volumetric pump (not shown) through the conduct 7, and then reaches the mixer 3 of the lowest stage, wherein it comes into contact with the brine; the soap and brine mixing is firstly performed within the mixer 3 of this lowest stage, and the mixture flows out through pipe 5 and reaches the lower settling compartment 4. The brine flows down into the lower part of this compartment 4 of the lowest stage, and is drawn off through the conduct 11 by a volumetric pump (not shown). The soap accumulates at the highest part of the compartment 4 of the lowest stage, and flows therefrom through a pipe 6 into the mixer 3 of the next and higher stage, wherein it comes into contact with the brine flowing from the compartment 4 of the next following and higher stage through pipe 8. Soap is so flowing up from stage to stage until the highest compartment 4 is full; it then flows into the vat 2, wherein a slightly alkaline water is urged through a pipe 9 and a volumetric pump (not shown); the temperature of this water is regulated by a thermostat 10 which acts upon the heating device (not shown) for the water; the mixer 2 makes the fitting easier by performing a close mixing of water with the soap previously treated with brine.

As already mentioned, the temperature of the arrangement and of the fluids flowing therethrough is maintained at 85° C. by any well-known suitable means, and the gases in the water as well as in the washing brine are removed preliminarily by any convenient means, not shown.

The tower 1 and the auxiliary devices may have a double outer wall within which a liquid at the temperature of 85° C. will be caused to flow; said liquid moreover may be conveniently the finishing water.

The finished soap flows through the pipe 12 into the kettle 13 which progressively fills up. The kettle 13 is cylindrical in shape and has a frusto-conical bottom wall formed with an angle of about 120°; the height of said kettle is made

slightly greater than its diameter and the opening 14 is arranged substantially at one fifth of the height of the kettle 13 below the top end thereof. Preferably the volume of the kettle 13 is substantially twenty times greater than the volume of soap produced by hour, whereby the finished soap becomes perfectly decanted in the upper part of the kettle 13 before reaching the pipe 14. Since in practice six hours only are required for the phases of the liquid soap to be separated, due to the physico-chemical conditions existing following the washing of said soap, it is thus apparent that in this manner a very large security is given for the obtention of a neat soap having good qualities.

When the kettle 13 becomes filled up, the pump P is started and an amount of mixture is drawn off the kettle 13 from the bottom thereof, which corresponds to a volume greater by 20% than the volume of nigre theoretically formed in the kettle 13.

At the same time, a more concentrated brine must be fed in the mixer tank 17; therefore the pipe 18 will be connected now with a source of more concentrated brine, having for instance a 15% concentration. Said more concentrated brine causes the mixture in the tank 17 to separate into neat soap and a poorer brine; according to my invention, the concentration of said poorer brine must be equal to 13.5% in the example described in order that it can be used for washing.

The arrangement is very easily controlled, by controlling the amount of finishing water which is added; said amount may be varied within a substantially large range, while giving yet sufficiently good results. Indeed, only the respective volumes of the phases in contact are changed, which is of no importance, since the volume of liquid drawn off the kettle 13 is maintained greater than the volume of nigre formed by about 20%. Generally speaking, a volume of 15 parts finishing water to every 100 parts soap gives satisfactory results.

As has been mentioned already, it is desirable to use a finishing water which has been made slightly alkaline by adding a caustic alkali in an amount of 0.2 to 2% by weight, according to the desired alkalinity for the finished product.

It is to be clearly understood that the embodiment shown and described has been given by way of illustration only, and that many changes could be made thereto without departing from the scope of my invention as defined in the appended claims.

What I claim is:

1. In a method for continuously purifying a totally saponified glycerinated soap obtained in the most concentrated liquid phase known as the neat soap, including washing said soap in counter-current with a first brine constituted by an alkaline metal chloride having a concentration slightly above the critical concentration below which said purified soap dissolves, the steps comprising, mixing said soap with water after washing with said first brine, circulating said mixture of soap with water in a settling zone where separation into neat soap and nigre takes place, drawing off continuously from said settling zone, at the bottom thereof a quantity of liquid greater than the quantity of said nigre, and at the top thereof the surplus of the purified neat soap, circulating said drawn off liquid containing nigre through a supplementary mixing zone with a second brine having a concentration

greater than that of said first brine and adjusted to produce after mixing a change of structure resulting in a mixture of deglycerinated neat soap and of a brine constituting said first brine and circulating said mixture of said first brine and deglycerinated neat soap for the washing of said glycerinated neat soap.

2. In a method for continuously purifying a totally saponified glycerinated soap obtained in the most concentrated phase known as the neat soap phase, including washing said soap in counter-current at a temperature between about 80° and about 90° C. with a first brine constituted by an alkaline metal chloride solution having a concentration slightly above the critical concentration below which said purified soap dissolves, the steps comprising, mixing said soap with water after washing with said first brine, circulating said mixture of soap with water in a settling zone to separate the neat soap at the top from the nigre at the bottom, continuously drawing from the bottom of the settling zone a quantity of liquid greater than the quantity of the nigre and continuously drawing from the surplus of said purified neat soap, mixing said drawn off liquid from the bottom of said settling zone with a second brine having a concentration greater than that of said first brine, the concentration of said second brine being so adjusted to produce in the mixture a change of structure to form deglycerinated soap and residual brine of the same concentration as said first brine and circulating the last named mixture for the first brine washing of the glycerinated neat soap.

3. In a method for continuously purifying a totally saponified glycerinated soap obtained in the most concentrated phase known as the neat soap phase including washing said soap in counter-current at a temperature between about 80° and about 90° C. with a first brine constituted by an alkaline metal chloride solution having a concentration slightly above the critical concentration below which said purified soap dissolves, the ratio of the volume of said first brine to the fatty acids in said soap being within the range of 30/70 to 50/70, the steps comprising, mixing said soap with water after washing with said first brine, circulating said soap in admixture with water in a settling zone, continuously separating the neat soap from the top of the settling zone and continuously drawing off a quantity of liquid from the bottom of the settling zone which is greater than the quantity of nigre at the bottom of the settling zone, mixing the drawn off liquid from the bottom of the settling zone with a second brine having a concentration greater than that of said first brine, the concentration of said second brine being so adjusted to produce a change in structure in the mixture to form deglycerinated soap and a residual brine of the same concentration as said first brine, and circulating the last named mixture for the first brine washing of the glycerinated neat soap.

4. In a method for continuously purifying a totally saponified glycerinated soap obtained in the most concentrated phase known as the neat soap phase including washing said soap in counter-current at a temperature between about 80° and about 90° C. with a first brine constituted by an alkaline metal chloride solution having a concentration slightly above the critical concentration below which said purified soap dissolves, the steps comprising, mixing said soap with a dilute aqueous solution containing from about 0.2 to about 2% of caustic alkali after washing with said first brine, circulating said

soap in admixture with said dilute aqueous solution of caustic alkali in a settling zone, continuously separating the neat soap from the top of the settling zone and continuously drawing off a quantity of liquid from the bottom of the settling zone which is greater than the quantity of nigre at the bottom of the settling zone, mixing the drawn off liquid from the bottom of the settling zone with a second brine having a concentration greater than that of said first brine, the concentration of said second brine being so adjusted to produce a change in structure in the mixture to form deglycerinated soap and a residual brine of the same concentration as said first brine, and circulating the last named mixture for the first brine washing of the glycerinated neat soap.

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