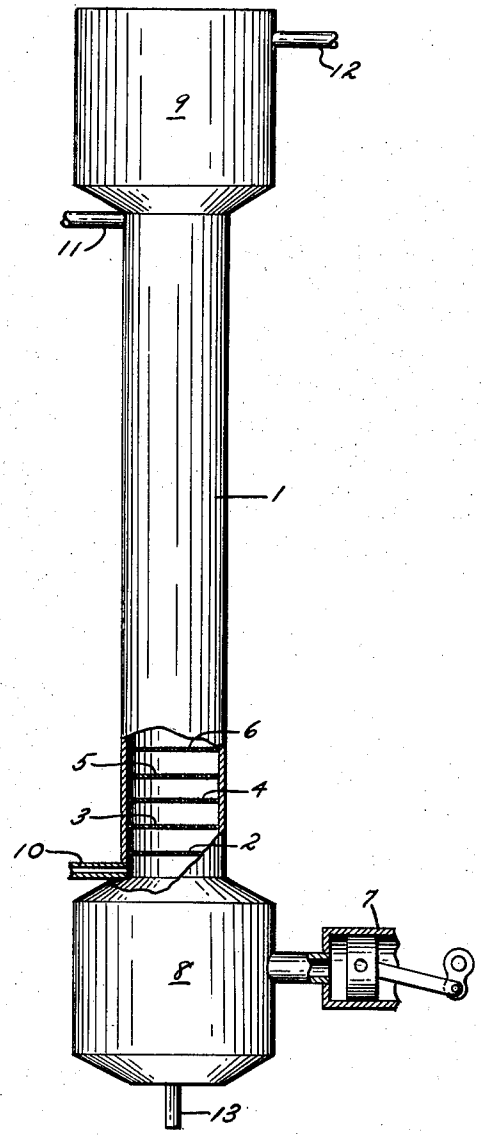
June 22, 1965 P. GODET ETAL 3,190,900

CONTINUOUS PROCESS FOR SOAP WASHING

Filed March 12, 1962



INVENTORS
PIERRE GODET
RENÉ GACHON
BY Michael J. Pantuliano
ATTORNEY

United States Patent Office 3,190,900 Patented June 22, 1965

3,190,900

CONTINUOUS PROCESS FOR SOAP WASHING

Pierre Godet, L'Isle-Adam, and René Gachon, Saint-Germain-en-Laye, France, assignors to Colgate-Palmolive Company, New York, N.Y., a corporation of Delaware

Filed Mar. 12, 1962, Ser. No. 178,829
Claims priority, application France, Mar. 23, 1961, 856,582
12 Claims. (Cl. 260—419)

The present invention relates to the manufacture of soap and more particularly to the extraction of glycerol and impurities from soap produced by the saponification of fats and oils.

The first stage of soap manufacture consists of a chemical reaction called saponification. The products of this reaction are soap and glycerol. However, this crude mixture also contains various impurities resulting either from the raw materials used or from secondary reactions accompanying the saponification reaction.

The crude soap must, therefore, undergo washings in order to remove to glycerol and the impurities. These washings are generally made with alkali or alkaline lye solutions whose electrolyte concentration is higher than the concentration at which the soap begins to be soluble in the lye. This is called "the limit lye."

Various authors have proposed processes permitting continuous washing, the latter presenting considerable advantages over the conventional kettle process. However, substantially all of these prior art continuous processes are derived from the soap kettle method, as they usually involve separating the two elementary operations performed in the kettle during a washing, that is, the mixing of the soap to be washed with a washing lye and the separating of the washed soap from the lye.

These continuous processes do not differ between themselves except by the type of apparatus used for making the mixtures (pumps, agitators, etc.) or the separations (settling tanks, centrifuges, etc.)

Although the soap washing problem, that is, the separation of glycerol from soap, is related to the conventional liquid-liquid extraction problem, the use of countercurrent extraction, in a washing column, until now has never been applied. The most closely related solution, although it still contains different stages of mixing and separating, is the procedure proposed by Monsavon, as described in the: Progress in the Technology of Fats and Other Lipids, vol. V, Advances in Technology, Pergamon Press, 1958, Chapter 2, Monsavon Continuous Process for Soap Manufacture, F. Lachampt, R. Perron.

However, the authors of this article emphasize the impossibility of operating according to the conventional liquid-liquid extraction process, when they write:

"Contrary to what would be expected, experience shows that although soap washed with limit lye settles rapidly, a simple column with countercurrent circulation of soap and lye will not suffice. The lye does not circulate by gravity in such a case. In fact, it is necessary to place pumps on each stage in order to move the lye in forced circuit."

Tests have confirmed this basic difference between the washing of soap to extract glycerol, and conventional liquid-liquid extraction. No success was achieved by trying to circulate the soap and the lye countercurrently in a column containing packing bodies; the two phases did not advance countercurrently, for the soap was retained by the packing bodies. On the other hand, it was also ascertained that by reducing the packing to a minimum in order to make possible the advance of the phases, practically no exchange of the glycerol from the soap to the lye occurred.

In essence, this is due to the fact that the soap phase cannot be considered as a liquid containing glycerol in solution. According to Wigner (S. H. Wigner, The Soap Manufacturer, the Chemical Processes, Chemical Publishing Co., New York, 1940):

"The soap phase is in fact formed by a pure soap hydrate containing 66% of fatty acids and a lye identical in composition to the one which separates from it."

The glycerol which has to be extracted by washing is not retained in solution in the soap phase, but is dissolved in the lye mixed with the soap. In other words, a division coefficient of the glycerol between the soap phase and the lye phase does not exist due to the fact that the amount of glycerol retained in the soap depends on the quantity of lye that it retains, and which according to the conditions is highly variable.

Therefore, taking into account the teachings of the prior art, it seemed impossible to perform the washing of the soap in a countercurrent column.

It has now been surprisingly discovered that passing soap to be washed and a washing solution through a pulsed column in countercurrent relationship to each other will effect the countercurrent washing of the soap by the lye or washing solution.

In this regard, it has been found that if the soap phase obtained after saponification is divided into very small grains, their coalesence into a single body does not occur before the passage of a relatively long time, i.e., approximately 30 minutes, even if these grains are brought into contact. This time has been found to be sufficient to permit the replacement of the glycerin-like lye, contained in the soap grains which are to be washed, by fresh lye, thus extracting the glycerol and the impurities which accompany it from the soap grains. The size of the grains of soap will determine the ease of the lye replacement operation. Preferably, the grains should have a volume of less than 100 cubic millimeters.

According to this invention, a process for extracting impurities and glycerol from "crude" soap comprises introducing countercurrently a washing lye and a crude soap containing said impurities and glycerol into a washing zone, pulsing the washing lye-crude soap mixture in the washing zone while simultaneously reducing and expanding cross-sectional areas of the washing lye-crude soap mixture at intervals along the length of the washing zone, and collecting the washed soap and the washing lye containing said impurities and glycerol in separately spaced settling zones.

More specifically, the instant invention relates to a continuous soap washing process, which comprises introducing continuously and countercurrently a washing lye phase and a crude soap phase containing impurities and glycerol into a column equipped with perforated plates arranged in tiers, the crude soap forming the upwards flowing phase, setting the liquid in the column into reciprocating motion adapted to effect the passage of the soap phase through the perforations of the plates in the form of small grains or droplets thereof, and continuously collecting washed soap and washing lye containing substantially all of said soap impurities and glycerol in settling zones disposed at the top and the bottom of the column respectively.

The function of the perforated plates in the practice of this invention is believed to be basically different from the function which they play in a liquid-liquid extraction column. In the latter instance, they are redispersion elements of a dispersed phase which has a tendency to coalesce quickly. In the present invention, the function of the plates is to break and to deform increasingly the soap grains in order to remove the lye impurity and to replace it with a washing lye having a smaller content of im-

purities and glycerol. In the pulsed column cross-sectional areas of the washing lye-crude soap mixture are simultaneously reduced (in the perforated plates) and expanded (in the zones between the perforated plates).

The advantages offered by this process and the apparatus employed for carrying out the process are numerous and significant. For example, the apparatus of the invention does not require the use of a circulation pump between the entrance and exit of soap, or any mixing device. Moreover, the perforated plates are permanent, simple, non-mechanical devices which do not require any maintenance.

However, while the aforementioned apparatus does not require any pumping or mixing device in order to insure the circulation and the contact of the products, it is to be understood that this apparatus constitutes only a preferred means of embodying the invention, and that one or several mixing or pumping devices may, if desired, be included in a particular embodiment of the apparatus, without, for this reason, departing from the scope of the invention.

Another advantage of the process of this invention resides in the fact that the apparatus is entirely closed. This is particularly advantageous with respect to the quality of the soap obtained, since no oxidation can take place by contact with air. Since the materials remain in the column only a very short time, it is not necessary to heat the column and, therefore, the steam consumption of the process is accordingly reduced to a minimum. Moreover, the simplicity of operating a column requires only a minimum of labor. Even manual intervention is not required as a simple and conventional automatic control can be easily adopted.

Finally, heretofore soap purification had to be made with lyes, the electrolyte concentration of which had to be as low as possible, the lower limit being the "limit lye." However, in this process, the concentrations of lyes can be maintained very near the "limit lye" and even slightly below, the upward velocity of the soap preventing the saturation of the lyes and, therefore, the formation of a nigre.

In the attached drawing, which is given solely as an illustration of the practice of this invention, the single figure represents schematically in elevation with partial sectioning, a column designed for carrying out the process of this invention.

The contents of the column whose body **1** is prolonged at each end by settling sections **8** and **9**, is pulsated by means of a device **7** which can be a piston pump (as shown), a pneumatic device or any other device imparting to the materials contained in the column an alternative or reciprocating motion upwards and downwards.

The body of the column comprises in the interior, horizontal plates or discs **2**, **3**, **4**, **5**, **6**, etc., perforated with holes or openings. The diameter of the holes can vary from 1 to 10 mm., but preferably should be from 2 to 6 mm. The number of holes per unit of surface of the discs can vary in such a way that the percentage of free passage through a disc comprises between 20 and 50%.

The spacing between the discs depends on several factors such as the required efficiency, the flooding limits, the pulsation characteristics, etc. This spacing can preferably vary from 2 to 20 cm.

The diameter of the column depends on the required production capacity. Different inlet and outlet pipes are connected to this column, as can be seen hereafter in the explanation of the operation.

The soap to be washed, coming from the saponification stage, is introduced in inlet **10**. In inlet **11** the washing lye is introduced, the composition of which as regards caustic and salt contents is such that the soap coming out at the top has the desired minimum content of cautsic and salt.

The soap introduced in conduit or inlet **10** thus flow upwards from inlet **10** to outlet **12** and meets the lye entering at inlet **11** and discharging at outlet **13**, the latter thereby washing the soap countercurrently.

Settling section **8** allows separation, through settling, of the lye from the soap which is to be washed, this lye being removed at outlet **13**. The settling section **9** is designed to separate the lye from the washed soap. The latter is removed at outlet **12**.

The volumes of the settling sections are such that they allow a sufficient settling time for the separation of the lye from the soap. This settling time can vary from 10 to 60 minutes. The diameter of the settling sections should be preferably larger than that of the column, for example, from 10 to 50% larger, as is customary in the construction of extraction columns.

Pulsation, i.e. the reciprocating displacement, imparted to the liquid of the column by the piston device **7** is characterized by its frequency and the amplitude of displacement of the liquid in the column. Expedient values for these characteristics have been found to be as follows:

(1) Frequency from 50 to 300 strokes per minute, but preferably 100 to 200.

(2) Amplitude from 2 to 50 mm., but preferably from 5 to 30 mm.

For the practice of this invention, the term "amplitude" may be defined as follows:

$$\text{Amplitude} = \frac{\text{volume of materials displaced by each stroke of the piston in cubic millimeters}}{\text{area of the cross-section of the column in square millimeters}}$$

Among the advantages which result from this working method, the following can be indicated:

(a) The possibility of reducing to a minimum the ratio of washing lye to soap, that is, the glycerol concentration in the lyes can be greater and, consequently, a saving on the subsequent concentration of the lyes necessary to recover the glycerol can be effected.

This can usually be done in any process by increasing the number of washing stages but, in fact, due to the cost of equipment necessary for the realization of the additional stages, as for example, the cost of the installation of pumps, settling tanks, centrifuges, etc., these stages are actually limited to 3 to 6. On the other hand, using the process of this invention, merely increasing the length of the pulsed column is sufficient to increase the number of theoretical stages, and this at a very low expense.

(b) By virture of the fact that an entirely closed apparatus is employed, in which there does not exist intermediary settling zones requiring a settling of fluids in order to carry out a separation, electrolyte concentrations in the washing lye may be employed as close as is desired to the limit lye, and even slightly below it. This feature is especially favorable for the purification of soap.

(c) The composition of the washed soap coming out from the column is remarkably constant, and therefore permits a soap of a constant quality to be obtained.

The following illustrative examples further indicate the practice of this invention.

Example 1

A 120 cm. diameter column, having a configuration as illustrated in FIG. 1, 5.7 meters high and whose packing was formed by 46 discs perforated with 50,000 3 mm. holes, was pulsated at the rate of 150 strokes per minute with an amplitude of 6.5 mm. of displacement of liquid in the column.

This column was fed at the base with 6000 kg. per hour of sodium soap obtained through saponification of a mixture of 85% tallow and 15% coconut oil at a temperature of 80 to 100° C. The soap contained 3.7% glycerol. Three thousand (3,000) kg. per hour of a washing lye was fed into the column at a temperature of from 80 to 100° C. This lye contained 6.3% salt and 2.1% caustic soda.

The electrolyte concentration of this lye was lower than the limit lye. This was observed from the appearance of the lye.

At the upper exit of the column a soap containing only 0.1% glycerol was obtained.

Example 2

A 40 cm. diameter column, having a configuration as illustrated in FIG. 1, 4.8 meters high whose packing was formed by 28 discs perforated with 4000 4 mm. holes, was pulsated at the rate of 180 strokes per minute with a 10 mm. displacement amplitude of the liquid in the column.

This column was fed at the lower inlet with 650 kg. per hour of sodium soap, obtained through saponification of a mixture of 40% tallow, 12% coconut oil, 36% fat and 12% rosin at a temperature of 80 to 90° C. The soap contained 4% glycerol.

Washing lye was fed into the column at the rate of 350 kg. per hour through the upper inlet. This lye contained 8.2% salt and 1.8% caustic soda. The electrolyte concentration of this lye was higher than the concentration of limit lye. The washed soap exiting from the top outlet of the column has a glycerol content of .2%.

Example 3

A 70 cm. diameter column, having a configuration as illustrated in FIG. 1, 4 meters high and whose packing was formed by 50 discs perforated with 5000 5 mm. holes was pulsated at the rate of 120 strokes per minute with a 5 mm. displacement amplitude of the liquid in the column.

This column was fed at the base with 2000 kg. per hour of sodium soap obtained through saponification of a mixture of 80% peanut oil and 20% coconut oil at a temperature of 90° C. This mixture contained 5% glycerol.

Nine hundred (900) kgs. per hour of a lye at 90° C. and containing 6.75% of salt and 1.75% of caustic soda were fed into the top inlet of the column. The electrolyte concentration of this lye was lower than the limit lye concentration.

The washed soap exiting from the top outlet of the column had a glyceral content of 0.4%.

Example 4

A 50 cm. diameter column, having a configuration as illustrated in FIG. 1, 7 meters high and whose packing was formed by 45 discs perforated with 2650 6 mm. holes was pulsated at the rate of 200 strokes per minute with a 3 mm. displacement amplitude of the liquid in the column.

This column was fed at the base with 1000 kgs. per hour of a sodium soap obtained through saponification of a mixture of 60% tallow, 20% palm oil and 20% cabbage palm oil at a temperature of 95° C. This soap contained 5.7% of glycerol.

Six hundred fifty (650) kgs. per hour of a lye at 100° C. and containing 5.85% of salt and 1.90% of caustic soda were fed into the top inlet of the column. The electrolyte concentration of this lye was lower than the limit concentration.

The soap coming out at the upper part of the column contained 0.07% glycerol.

Example 5

A 50 cm. diameter column, 5 meters high and whose packing was formed by 40 discs perforated with 28,000 3 mm. holes was pulsated at the rate of 100 strokes per minute with a 7 mm. displacement amplitude of the liquid in the column.

This column was fed at its lower inlet with 3500 kgs. per hour of a potassium soap at 90° C. obtained through saponification of olive oil. This soap contained 5.2% glycerol.

The washing lye fed into the column at the rate of 1800 kgs. per hour through the upper inlet of the column was a solution containing 12% of potassium chloride. This concentration was higher than the limit lye concentration.

The soap washed at the head of the column did not contain more than 0.20% glycerol.

It is to be understood that the invention is not limited to the aforementioned specific process and apparatus. Various changes can be made in the arrangement, form, construction and type of the various elements of the apparatus and various modifications can be made in the process without departing from the spirit or scope of the invention.

What is claimed is:

1. A process for extracting impurities and glycerol from crude soap which comprises introducing countercurrently a washing lye and a crude soap containing said impurities and glycerol into a columnar washing zone, said crude soap forming the upwards flowing phase, pulsing the washing lye-crude soap mixture in the washing zone to impart a reciprocating motion to said mixture while simultaneously reducing and expanding cross-sectional areas of the washing lye-crude soap mixture at intervals along the length of the washing zone by means of a plurality of perforated plates arranged in tiers, said soap of said mixture being thereby reduced in particle size and collecting washed soap and washing lye containing substantially all of said impurities and glycerol in separately spaced settling zones disposed at the top and the bottom respectively of the washing zone.

2. A continuous process for extracting impurities and glycerol from crude soap which comprises continuously introducing countercurrently a washing lye and a crude soap containing said impurities and glycerol into a columnar washing zone, the crude soap forming the upwards flowing phase; continuously pulsing the washing lye-crude soap mixture in the washing zone to impart a reciprocating motion to said mixture while simultaneously reducing and expanding cross-sectional areas of the washing lye-crude soap mixture at a plurality of intervals along the length of the washing zone by means of a plurality of perforated plates arranged in tiers, said soap of said mixture being thereby reduced in particle size, and continuously collecting washed soap and washing lye containing substantially all of said impurities and glycerol in settling zones disposed at the top and the bottom respectively of the washing zone.

3. A process according to claim 2, wherein the washing lye has a concentration of about the limit lye.

4. A process according to claim 2, wherein the washing lye has a concentration of slightly below the limit lye.

5. A continuous process for extracting impurities and glycerol from crude soap which comprises introducing continuously and countercurrently a washing lye phase and a crude soap phase containing said impurities and glycerol into a washing zone fitted with a plurality of perforated plates arranged in tiers, the crude soap forming the upwards flowing phase, pulsing the washing lye-crude soap mixture in the washing zone to impart a reciprocating motion to said mixture and to effect passage of the soap phase through the perforations of the plates in the form of small grains thereof, and continuously collecting washed soap and washing lye containing substantially all of said impurities and glycerol in settling zones disposed at the top and bottom respectively of the washing zone.

6. A process according to claim 5, in which the grains of soap passing through the holes of the perforated plates are reduced in size to a volume of less than 100 cubic millimeters.

7. A process according to claim 5, wherein the washing lye has a concentration of about the limit lye.

8. A process according to claim 5, wherein the washing lye has a concentration of slightly below the limit lye.

9. A process according to claim 5, wherein the perforated plates arranged in tiers spaced 2 to 20 cm. apart holes of said plates have a diameter of from 1 to 10 mm.

10. A continuous process for extracting impurities and glycerol from crude soap which comprises introducing continuously and countercurrently a washing lye phase and a crude soap phase containing said impurities and glycerol into a washing zone fitted with a plurality of perforated plates arranged in tiers spaced 2 to 20 cm. apart and containing holes having diameters of from 1 to 10 mm., the soap forming the upwards flowing phase, animating the washing lye-crude soap mixture with a reciprocating displacement of from 2 to 50 mm., said reciprocation having a frequency of from 50 to 300 per minute, and continuously collecting washed soap and

washing lye containing substantially all of said impurities and glycerol in settling zones disposed at the top and bottom respectively of the washing zone.

11. A process according to claim 10, wherein the diameters of the holes of the perforated plates are from 2 to 6 mm.

12. A process according to claim 10, wherein the displacement is from 5 to 30 mm. and the reciprocation has a frequency from 100 to 200 strokes per minute.

References Cited in the file of this patent

UNITED STATES PATENTS

2,562,207 Owen ---------- July 31, 1951

UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

Patent No. 3,190,900 June 22, 1965

Pierre Godet et al.

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 1, line 22, for "to", second occurrence, read -- the --; column 3, line 73, for "cautsic" read -- caustic --; line 74, for "flow" read -- flows --; column 4, line 48, for "virture" read -- virtue --; column 5, line 44, for "glyceral" read -- glycerol --; column 7, line 2, for "arranged in tiers spaced 2 to 20 cm. apart" read -- are spaced from 2 to 20 cm. apart and the --; column 8, line 9, after "frequency" insert -- is --.

Signed and sealed this 26th day of April 1966.

(SEAL)
Attest:

ERNEST W. SWIDER
Attesting Officer

EDWARD J. BRENNER
Commissioner of Patents