# United States Patent [19]

**Smith et al.**

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[54] **SUPERFATTED SOAP AND PROCESS OF PRODUCING IT**


[75] Inventors: **Brian Robson Smith**, Chadderton; **Kenneth Tomlinson**, Bramhall, both of England

[73] Assignee: **Colgate-Palmolive Company**, New York, N.Y.



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[63] Continuation of Ser. No. 366,546, June 3, 1973, abandoned, which is a continuation of Ser. No. 167,877, July 30, 1971, abandoned, which is a continuation of Ser. No. 730,634, May 20, 1968, abandoned.

[52] **U.S. Cl.** ..... **252/132;** 252/108; 252/134; 252/368; 252/370; 252/DIG. 16

[51] **Int. Cl.²** ..... **C11D 9/48;** C11D 11/04; C11D 13/00

[58] **Field of Search** ..... 252/108, 132, 367, 368, 252/369, 370, DIG. 16, 134



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*Primary Examiner*—Dennis E. Talbert, Jr.
*Assistant Examiner*—Dennis L. Albrecht
*Attorney, Agent, or Firm*—Herbert S. Sylvester; Murray M. Grill; Norman Blumenkopf



[57] **ABSTRACT**

Superfatted soap, especially toilet soap bars, comprising the alkali metal, preferably sodium, salts of fatty acids, free fatty acids in the range of about 4% to 15% by weight of the total fatty acid content of the soap, and an electrolyte content of less than about 1% by weight of the soap. The soap is produced by fully saponifying the fatty charge, e.g., in a kettle by the kettle soap making process and working up the saponified mass to the fitting stage, adding sufficient acidifying agent, e.g., a mineral acid and preferably hydrochloric acid, to liberate from about 4% to 15% of fatty acids based on the total fatty acid content, separating the acidified mass into neat soap and nigre, and working up the neat soap into bars.


**3 Claims, No Drawings**

# SUPERFATTED SOAP AND PROCESS OF PRODUCING IT

This is a continuation of application Ser. No. 366,546, filed June 3, 1973 now abandoned, which is a continuation of Ser. No. 167,877, filed July 30, 1971, and now abandoned, which is a continuation of Ser. No. 760,634, filed May 20, 1968, and now abandoned.

## DESCRIPTION

The present invention relates to superfatted soap and a process of producing it. More particularly the soap is superfatted by the presence in it of free fatty acids derived from salts of fatty acids in the soap and the electrolyte content is that normally present in properly fitted neat soap. The soap is preferably of toilet quality in the form of tablets or bars. The process of making the soap according to the invention follows any suitable soap making process to the fitting stage when an acidifying agent is added in sufficient quantity to free the desired proportion of the fatty acids from the salt form. Preferably hydrochloric acid is used because the product of the reaction of the acid with the soap produces alkali metal chloride, usually sodium chloride, which is the electrolyte already present in the soap mixture from the salt washes, but other acidifying agents such as other mineral acids may be used. The acidified mixture is then fitted, i.e., a separation of neat soap from nigre is effected, and the neat soap is worked up in the usual manner into bars.

Suitable soap making processes include the usual kettle soap making process and continuous saponification processes in which saponification is effected with aqueous alkali and heat, the resultant mixture being washed and fitted to obtain a neat soap either continuously or by use of a kettle for the fitting and optionally one or more washing stages.

The kettle process of making soap is well known to those skilled in the soap making art so it will be disclosed here only in general outline since it does not form part of the present invention. A typical kettle process includes the following stages:

1. Killing or saponification stage.
2. Change stage.
3. Washing stage.
4. Fitting or pitching stage.

In the first or saponification stage the fatty material chosen for the particular soap to be produced, e.g., fats and oils which are glyceryl esters of fatty acids, is heated with aqueous caustic solution to effect substantial conversion of the fatty material to glycerine and fatty acid salts of the alkali metal or metals present in the caustic solution. For toilet bars sodium soaps are generally preferred but in some cases caustic potash may be used with caustic soda in the caustic solution. Potassium soaps are softer than sodium soaps of the same fatty acid content and therefore the content of potassium fatty acid salts is somewhat limited in proportion to the sodium fatty acid salts in order to have a soap that works up well into bar form in conventional soap making machinery. Generally, the caustic solution used in the killing stage is obtained in whole or in part from the change stage of another batch. The extent of saponification in the killing stage is not critical and may vary from as low as about 80% to almost complete saponification.

In the change stage, which may take place in one or more separate operations, salt is added to the reaction



mixture in sufficient concentration to effect precipitation of the soap from the solution. The salt used may be fresh salt or salt recovered from the purification of the glycerine, or a mixture of both. Generally, boiling is continued for a short time after addition of the salt and if the saponification is incomplete in the killing stage caustic may be included with the salt to complete the saponification during this boil. The mass is then settled, during which the less dense soap rises to the top and the spent lye containing the bulk of the glycerine and electrolyte sinks to the bottom. When the settling is complete the lye is drawn off and sent to the glycerine plant for recovery of the glycerine, salt, etc.

The washing stage has as its object the more complete removal of glycerine, which may be as much as 0.5% of the weight of the soap layer left after drawing off the lye, and the complete saponification of the fat charge. In general, a washing operation consists of closing the soap by adding water and boiling vigorously, usually with open steam, and then adding salt and/or caustic to grain the soap and permit settling again into an aqueous lower layer and an upper grained soap layer. Where caustic is used in the washing operation it is sometimes called a strengthening change and the lye settled is not fully spent and may be used as the caustic solution in the killing stage of another batch of fatty material. The washing operation also removes further amounts of impurities that may have been present in the original charge.

The fitting or pitching stage has for its objective the separation of a neat soap phase from a nigre phase and it is effected by again closing the soap by the addition of water and boiling. The water addition is carefully done so as to reach a final stage where the electrolyte content of the mixture is sufficient to dissolve part of the soap but not all of it. On settling the dissolved soap sinks to the bottom and forms the nigre while the undissolved soap rises to the top as neat soap.

A typical composition of the soap mass prior to fitting is approximately 58% to 59% total acids, 0.16% to 0.2% caustic calculated as $Na_2O$ and 0.7% to 0.9% sodium chloride, with water at about the 30% level. It is at this stage of the soap making process that the process is modified according to the invention by acidifying the mass prior to the fitting operation. This may be and preferably is done by adding hydrochloric acid to liberate some 4% to 15% of the fatty acids based on the total fatty acid content of the mass.

Considerable variation is permissible in the fitting operation within the scope of the invention. For example, instead of adding water only to effect the formation of the nigre, a solution of salt can be used which usually results in the formation of a larger nigre layer but the finish point is approached more slowly and a more consistent finish can be obtained. Another variation is to add some water to form an open finish, allow the mass to settle for a while and remove the nigre formed, then reboil and finish with salt solution which allows the accurate finish of the salt solution method but does not have the disadvantage of a large nigre formation. The acid can be added at any stage of a finishing operation, i.e., after the last of the washing operations.

The acid performs two functions:

1. It reacts with any free alkali to form additional salt. Salt is required to form the nigre and approach a consistent finish.

2. It reacts with the soap to form the free fatty acids desired for superfatting, and some salt.

If the fitting is performed with a single nigre separation, the acid is added after the last wash and it is usually necessary to add only water for the fitting because of the formation of salt in the mass by the addition of the acid. If there is a preliminary removal of the first nigre, then the addition of acid is preferably made after this separation and again only water would usually be added because of the formation of salt by the reaction of the acid with the soap and in actual practice the water may desirably be added with the acid in the form of a dilute HCl solution. The specific method chosen will depend on various factors such as the amount of free fatty acid desired in the final composition and the actual details of the kettle house system. If more than about 5% free fatty acids are desired, the acidification may be carried out in two steps, e.g., after the final wash acid is added in an amount sufficient to form all but about 3% of the desired free fatty acid which forms enough salt to grain the soap which is allowed to settle and the lye is removed. The remaining soap is then treated first with acid to form the final required free fatty acid and then with water to bring the kettle to a finish. Either one or two nigres may be removed in this procedure.

A typical composition of the acidified mass prior to fitting is approximately 56% total fatty acids, about 4% to 15% free fatty acids (FFA) based on the total fatty acid content, i.e., about 2.2% to 8.4% free fatty acids based on the acidified mass, 1.3% to 1.6% sodium chloride, no free caustic and about 30% water. After fitting, the typical compositions of the two phases formed on settling are approximately:

| | Neat Soap | Nigre |
|---|---|---|
| TFA | 62% – 63% | 25% |
| FFA | 2.4% – 9.5% | 0.5% – 2.0% |
| $Na_2O$ | nil | nil |
| NaCl | 0.5% | 5% – 7% |

The ratio of neat soap to nigre is about 5:1 and unexpectedly, the free fatty acids produced do not divide equally between the neat soap and nigre phases but remain preferentially in the neat soap phase. The actual ratio of the concentrations of FFA in the neat and nigre phases is about 5:1.

Similar results may be obtained by other soap making processes, such as the continuous processes referred to above. These processes are also well known to workers of ordinary skill in this art. The novel operations of the present invention are (1) acidification of fully saponified soap just prior to fitting and (2) fitting the acidified soap to obtain separation of neat soap from nigre, the neat soap then being worked up into bars in any desired way.

It has been proposed heretofore to produce a superfatted soap by adding hydrochloric acid to a fully saponified mixture of coconut fatty acids and colophony or castor oil in a proportion of about 100 parts of fatty acids to 5 parts by weight of colophony or castor oil, the amount of acid being about 7 parts of 30% HCl. The acidified mixture is then worked up in bar form in the usual way, i.e., by crutching, framing and cutting. This process leaves the salt formed in the acidification step in the soap, which can be tolerated in a soap made largely from coconut oil fatty acids but it is uneconomical to make soap of this composition because of its higher cost than tallow. Such a soap also has the disadvantage of a high proportion of salts of lower fatty acids having from 6 to 10 carbon atoms per molecule which are known to be irritating to sensitive skin when present in soap. While coconut and like acids can be topped to remove these irritating lower acids, the removal operation adds considerably to the cost of soap production. The present invention, on the other hand, permits the use of all soap making fatty acids by eliminating all of the salt in excess of the normal content of properly fitted soaps.

It has also been proposed to make superfatted soaps by either incomplete neutralization of fatty acids used as the starting material in the soap making process, or by adding free fatty acids to fully saponified fatty material. The partial neutralization process results in a soap that does not contain salt which is a desirable ingredient, within critical limits, in a soap that is to be worked up by milling and plodding because it makes the soap harder and easier to work in the mill, the plodder and the press or stamp. The addition of free fatty acids to fully saponified fatty material requires a special preparation of these acids which adds cost to the product compared to the present process which forms the free fatty acids from the fat used in the kettle charge. The free fatty acids used in the neutralization process are also much more expensive than the fats and oils for the same amount of soap.

The fat charge used in the present invention is not critical, but it is preferred to use a charge comprising about 40% to 90% tallow and 10% to 60% of the oils of the coconut type, including coconut oil, palm kernel oil, Babassu oil, and the like. A soap made from this combination of raw materials by the process of the invention is a new composition of matter comprising the sodium salts of tallow and coconut oil fatty acids in the ratio of about 40% to 90% tallow and 60% to 10% coconut oil fatty acids, an excess of fatty acids in the range of 4% to 15% based on the total fatty acid content of the composition derived from fatty acids in the soap or salt form, and an electrolyte content of at least about 0.25% to less than about 1%, and is part of the present invention.

Fatty material that may be used in the fat charge to the kettle include the following:

| | |
|---|---|
| Coconut Oil | Castor Oil |
| Palm Kernel Oil | Groundnut Oil |
| Babassu Oil | Linseed Oil |
| Palm Oil | Cottonseed Oil |
| Animal Fats | Fish Oils |
| Olive Oil | and hydrogenated versions of |
| Tall Oil | these products |
| Rosin | |

The caustic used to saponify the fat charge is preferably sodium hydroxide but it may be replaced in part by potassium hydroxide, e.g., up to about 15% if desired.

It is advantageous to include a preservative in the soap to prevent the rancidification of the free fatty acids and among suitable preservatives are the following:

2–6 ditertiary butyl p-cresol, tertiary butyl hydroxyanisole, "Sopanox", triphenyl phosphite.

The usual steps for converting fitted soap into bar form include crutching the neat soap phase with desired additives such as preservatives and heavy metal sequestrants such as stannic chloride and ethylene diamine tetra-acetate, then drying the soap to a moisture content within the range of about 8% to 15%, prefera-

bly about 9–10%, mixing the dried soap in the form of dried ribbons, granules, or the like in an amalgamator with other desired additives such as colors, perfumes, glycerine, lanoline, cold cream, etc., milling the mixture, plodding the milled chips to form a long, continuous bar, cutting the bar into tablet or cake lengths and pressing or stamping the tablet or cake which may then be wrapped or not as desired.

The following example illustrates the process and product of the invention: The fat charge consisting of 70 parts tallow and 30 parts coconut oil is fully saponified by boiling in a kettle at a temperature of about 105°C. for about 4 hours. After the customary washing stages, hydrochloric acid is added in sufficient proportion to produce about 5% FFA in the final soap. The acidified mixture is then fitted to obtain a separation of neat soap from a nigre. The neat soap is then worked up by crutching, drying, amalgamating, milling, plodding, cutting and pressing in the usual way.

The composition of the final bar comprises the following ingregients in approximately the indicated percentages:

| | |
|---|---|
| Soap | 85.0% |
| Moisture | 8.0% |
| FFA | 5.0% |
| Perfume | 1.0% |
| NaCl | 0.5% |
| Preservative, color, etc. | 0.5% |

Soaps produced by the process described are greatly superior to the corresponding soaps produced without acidification. The volume of lather produced is greater, and the lather tends to be rich, thick and creamy in texture. The concentration of soap in the lather is lower than with the corresponding unacidified soap, and consequently the loss of material from the bar is less. This makes soap made by the above process more economical in use than the corresponding soap made without acidification.

The process of acidification does not free the various fatty acids in the proportions in which they were present in the original soap. Acidification pushes out preferentially the shorter chain fatty acids in higher proportion than the longer chain fatty acids, and the unsaturated acids in higher proportion than the saturated acids. For these reasons, the process of the present invention produces a soap product of novel composition and properties which cannot be obtained except by the addition of acid prior to fitting and separation of neat soap from nigre as described.

Although the present invention has been described and illustrated with reference to certain specific embodiments, it is to be understood that variations and modifications can be made without departing from the spirit and scope of the invention as defined in the following claims.

What is claimed is:

**1.** The process of producing soap characterized by a higher ratio of saturated fatty acids to unsaturated fatty acids in a neat soap phase than in a nigre phase comprising:

1. adding to an alkali metal soap selected from the group consisting of sodium soap and a mixture of sodium soap and up to 15% potassium soap, said soap being obtained by saponifying a fatty mixture of 40–90% tallow and 10–60% coconut oil with sodium hydroxide and precipitating with salt, just prior to the fitting stage a sufficient quantity of hydrochloric acid to produce a free fatty acid content of about 4% to about 15% based on the total fatty acid content of said soap and to form a salt by neutralizing any remaining sodium hydroxide and
2. separating the acidified soap into neat soap and nigre, and working up the neat soap into bars.

**2.** The process according to claim **1** wherein step (2) is conducted by adding salt and water to the acidified soap and boiling same, the amount of water being sufficient to dissolve a part but less than all of the soap mass, to produce an undissolved neat soap phase and a dissolved nigre phase.

**3.** The process according to claim **2** wherein the nigre phase is removed and the remaining nigre free undissolved neat soap phase is reboiled and salt is added thereto.

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