

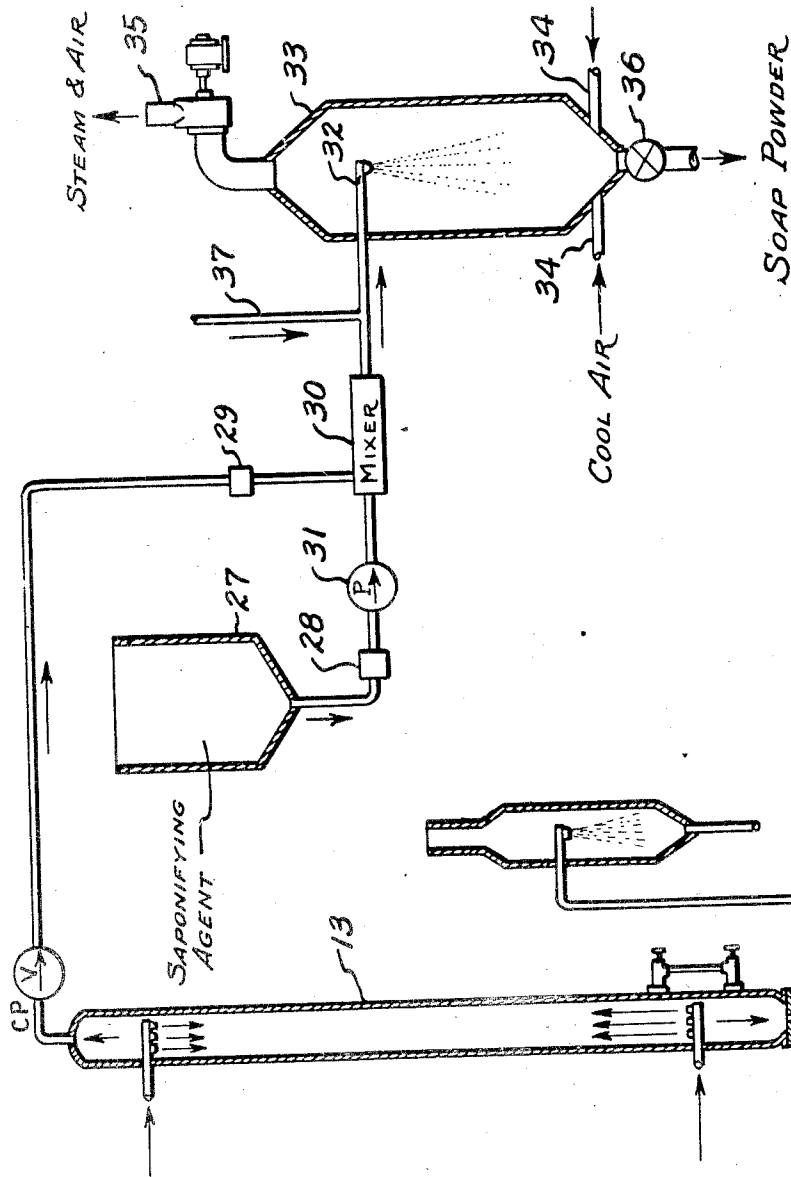
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SOLID COMMINUTED SOAP

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2,287,698

SOLID COMMINUTED SOAP

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1 Claim. (Cl. 252—368)

My invention relates to a new form of solid comminuted soap, and the process of preparing same.

The object of this invention is to provide comminuted soap having a new form with a combination of especially desirable properties including a solid structure (not hollow), absence of an overdried surface, a relatively high bulk density, a moderately high moisture content as compared to other forms of dried comminuted soap, rapid solubility in water without balling or lumping, and a fairly free flowing property.

This application is a continuation-in-part of my co-pending application Serial No. 64,525, issued May 23, 1939 as Patent No. 2,159,397 entitled Continuous process for converting saponifiable fats into soap and glycerin. My prior application discloses a particular method of preparing a comminuted soap having the above mentioned properties, but the present application is directed to converting soaps in general to the new form of solid comminuted soap, and particularly to the product itself.

Soap in comminuted form is becoming increasingly important to the soap maker as its advantages are realized more and more by the consumers. It advantageously displaces soap in form of bars and flakes, not only for household laundry use, dish washing, etc., but also for washstand toilet use in dispensers. The forms of comminuted soap heretofore marketed range from finely divided, dry, dense particles, slow in dissolving, such as powders for shaving and for use in dispensers, to relatively large sized hollow particles of very light weight, and very low moisture content, but quick to dissolve. A product that is of moderately heavy bulking weight, free from overdried or hardened surface, of rapid dissolving properties without balling or lumping, flowing freely from a container, and with moderately high moisture content as compared to ordinary spray dried soap products, is a desirable combination of properties which has not heretofore been met, but which is accomplished in the present invention.

The process and product described herein relate to comminuted soap either in pure form or containing only moderate proportions of soap builders, and in this respect it differs markedly from certain prior processes and products relating to "soap powder" which terms differs from "powdered soap" or comminuted soap, and is commonly used to indicate a mixture of soap with strongly alkaline ingredients of a crystalline nature such as soda ash in particular, in which the

proportion of such alkaline ingredients is very high and gives to the product a crystalline structure; soap powders furthermore usually contain large percentages of moisture, such as forty per cent or more. "Powdered soaps" or "comminuted soaps" on the other hand are free from a noticeable crystalline structure and always contain less moisture than that inherent in the soap base from which they are made, such as ordinary kettle soap which contains in the neighborhood of thirty per cent moisture.

The attached drawing shows diagrammatically an apparatus suitable for carrying out my process and making the product herein described, starting with fatty acids produced from fat in a continuously acting autoclave and making the soap by a continuous process. The method of operating will be apparent from the following description.

The fatty acids issuing from the continuous autoclave 13 (containing dissolved water in amount depending on the temperature and pressure), and having a temperature above 212° F., preferably about 470° F., are passed to mixer 30 through meter 29 while at the same time a solution of a saponifying agent, preferably caustic soda or soda ash, in amount to properly saponify the fatty acids, continuously controlled in relation to the fatty matter by a suitable proportioning device, is likewise passed continuously into the same mixer by pump 31 from tank 27 through meter 28. Meters 28 and 29 enable the operator to control the flow of saponifying agent and fat in proper proportions as desired, or they may be suitably connected with pump 31 in an automatic proportioning device. When a built soap product is desired, the solution of builder may be added to the solution of saponifying agent, or it may be added to the mixer separately in properly and continuously proportioned quantities. The total amount of water introduced in the mixer is so regulated as to give the desired moisture content in the final comminuted soap product, after allowing for evaporation in the spray chamber. The total amount of water in the soap should not be more than sufficient to form the "neat" soap phase, in order to assure ease of handling. No application of heat is necessary in the mixer or subsequently, because the fatty acids as received from the autoclave system already contain an adequate amount of heat for the subsequent operations, having a temperature preferably in the neighborhood of 470° F. The saponifying agent does not need to be heated more than enough to have it in good pumping condition but it may be

heated, if desired, to impart additional heat to the soap before spraying to secure maximum evaporation. The mixer must necessarily be kept under a sufficient pressure to prevent volatilization of the water at the temperature of mixing, and should be of sufficient size and capacity to keep the materials in contact with each other long enough to insure intimate contact and completion of the chemical reaction. This period need not ordinarily exceed one minute with an efficient mixer.

The hot soap mixture then passes directly to the spray nozzle 32 and issues therefrom into the spray chamber 33 in comminuted form. Inasmuch as the soap mixture reaches the nozzle at a temperature considerably above 212° F., usually about 250°-400° F., and under superatmospheric pressure, which may be as high as 100 to 550 pounds per square inch, the pressure drops suddenly to that of the spray chamber, usually atmospheric pressure, with a consequent drop in temperature and instantaneous evaporation of a portion of the water content in the soap. Cooling air is admitted at the bottom of the spray chamber through openings 34 in order to cool the product sufficiently to keep the particles from sticking together, and passes outward at the top through exhauster 35, thus incidentally carrying away the water vapor volatilized from the soap mixture. The soap mostly falls to the bottom of the chamber where it can be continuously removed through air lock 36 for packaging, or for further processing into other forms if desired. Any fine material which may pass out of the top of the chamber with the air is caught in a suitable dust collector.

Instead of making the soap continuously from fatty acids and saponifying agent as above described, it is also possible to use soap previously prepared in any other manner, in which case the said soap will be introduced through pipe 37 into the pipe leading to nozzle 32 without passing through mixer 30. In this case the soap, such as that described below, if not already within the desired temperature range of about 250° to about 400° F., is heated to a temperature within this range, preferably by pumping it through a heat exchanger supplied with a heating medium, such as steam or a mixture of diphenyl and diphenyl oxide known as Dowtherm, for example, while maintaining a pressure at least equal to the vapor pressure of the soap. I prefer to avoid volatilization of moisture at this stage, although this is not essential, and for this purpose I generally use an excess pressure of at least 100 pounds per square inch above the vapor pressure of the soap. The soap is then conducted to the spray nozzle 32 through which it is sprayed into a gas of lower pressure, usually into a current of cooling air. Suitable builders such as silicate of soda, soda ash, trisodium phosphate, borax, etc., may be added to the soap if desired and thoroughly mixed therewith before the soap is subjected to the spraying and cooling treatment. These materials may be added to the soap either in dry form or in form of a solution, but in the latter case care should be taken that the water thus added shall not be sufficient to convert the soap mass into a different phase such as the middle soap phase. The amount of builders thus added must in no case be sufficient to impart a crystalline structure to the product.

Soap suitable for use in the practice of the present invention may be any soap suitable for

use in water for detergent purposes, which is in the neat phase, but is solid at ordinary temperatures. Sodium soaps made from the common fats and oils, such as the soaps ordinarily used for toilet and laundry purposes may be used. It is important that the soap or soap mixture as used should be in the neat phase and in pumpable condition at the temperature of spraying; soap in the "middle" phase is too thick and gummy to pump, even though it contains more moisture than soap in the neat phase, and soap in the "nigre" phase, altho free flowing, contains too much moisture at the temperature used to permit of making the product of the present invention in the manner described. The soap used may be the ordinary kettle soap as made by the well known boiling and settling process, containing when finished about thirty per cent of moisture and having a temperature when freshly made and settled of about 180° to 200° F. Soap made by the "cold process" or "semi-boiled" process may also be used, but preferably when made from fatty stocks containing little or no glycerine.

The air supplied to the spray chamber may be introduced in either a concurrent or countercurrent direction with the soap, and by suitable adjustments of the moisture content and temperature of soap, and temperature and volume of air, practically any desired variation in moisture content of the product may be obtained. The higher the temperature, the more moisture will be volatilized in the spraying operation. By starting with soap containing thirty per cent or less of moisture, for example, and heating same to a temperature between about 250° and about 400° F., and with suitable adjustments of the temperature and volume of air, a product can be obtained having any desired moisture content between about twenty-five per cent and ten per cent, but ten per cent is approximately the lowest percentage readily obtainable in practical operation.

By proper selection of the spray nozzle and proper adjustment of same, a comminuted soap product may be obtained having predominantly any size particles desired, from that of a fine dust to very coarse particles.

The soap product obtained as described is novel as far as I know in consisting of feather-like, irregular shaped solid, not hollow, particles of size easily visible to the naked eye, and may contain practically any desired amount of moisture. The particles mainly sink in water, but form a bulk product having a bulk density usually of about .25 to .40 as compared with water, due to irregular structure of the individual particles which prevents the particles from packing tightly together. The product is very readily soluble, and fairly free flowing, non-dusting, and shows little or no tendency to lump or ball together in water. In these latter respects it compares favorably with spray dried products of the rounded, puffed, thin-wall type, such as those described in the patent to Lamont No. 1,652,900, which are noted for their ready solubility and their free flowing qualities, but which are light bulking and usually of low moisture content. My product as described dissolves in 10 to 15 seconds in the solubility test described in Lamont's Patent No. 1,652,900, as follows:

"... These figures on speed of solubility were determined by placing ¾ grm. of soap product in ½ liter of water at 110° F. and stirring the product with a tablespoon at the rate of 25 double

strokes per quarter minute. The container holding the soap and water was provided with an internal removable screen which was lifted out of the water at desired intervals, say 10 to 15 seconds, and if any soap remained undissolved it could be seen on the screen."

I attribute the rapid solubility of the particles of my preferred product, in spite of their solid condition, to the irregular, thin, lacy structure of the particles due to the bursting or explosive effect in their formation, and to the fact that they have not acquired a hard dried skin on the surface of the particles, such as would result from exposure to a highly heated drying gas. All the heat for drying my product is stored up within it under high temperature and pressure conditions before reaching the spray nozzle, so the drying occurs just as much in the interior of the particle as on the surface, and the surface is no drier than the interior of the particle.

As an example where I have prepared a dried filled soap, I used a soap made from a mixture of 80% tallow and 20% coconut oil which was saponified with sodium hydroxide solution in a kettle in the ordinary manner, and after salting out and settling as usual the finished kettle soap had a moisture content of approximately 30%. A mixture was then made consisting of 80% of this kettle soap with 20% of a builder consisting of silicate of soda containing 40% solid matter. This soap mixture, containing approximately 36% moisture, was heated to a temperature of 390° F. under a pressure approximating 400 pounds per square inch by passing the mixture through a suitable heat exchanger, not shown, and then through pipe 37 to the nozzle 32 in spray chamber 33 where the sprayed mixture fell through an ascending column of cooling air having an initial temperature of about 70° F. About 290 cubic feet of air were used per pound of product. The product then contained about 18% moisture and had a temperature approximating 100° F. It consisted of comminuted soap of a size readily visible to the naked eye; over 50% of the particles by weight remained on a 60 mesh sieve; the indi-

vidual particles were heavier than water, but in bulk the density was found to be .34 as compared to water; the product was substantially free from dust, did not ball in water, and was fairly free flowing. It readily dissolved within about 15 seconds when subjected to the Lamont solubility test described above.

The comminuted soap of the present process can not only be used and sold as such, but it may also be readily converted into other forms of soap products such as flakes, bars, or extruded forms. For this purpose it is only necessary to produce first the comminuted soap having the formula and moisture content desired, and then pass it while in a plastic state, through rolls to convert it into a flaked product, or through suitable rolls and pladders or other known equipment to convert the particles into the form of extruded bars.

Having thus described my invention, what I claim and desire to secure by Letters Patent is:

A free flowing, substantially non-dusting, comminuted soap product having a moisture content of about ten to about twenty-five percent, a bulk density of about .25 to about .40 as compared to water, and composed predominantly of particles easily visible to the naked eye, said particles being individual fragments resulting from the explosion of larger particles and being of feather-like, irregular shape, individually heavier than water, having a surface and a uniform solid interior of about equal hardness and moisture content and a uniform composition throughout any cross section of same and which when placed in distilled water at 110° F. in the proportion of $\frac{3}{4}$ gram of the soap product to $\frac{1}{2}$ liter of water and stirred with a tablespoon at the rate of 25 double strokes per $\frac{1}{4}$ minute substantially as described, dissolves substantially completely as evidenced by the absence of any substantial proportion of undissolved particles when the soap-containing water passes through a 60 mesh sieve, in a period of time not substantially exceeding 15 seconds.

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