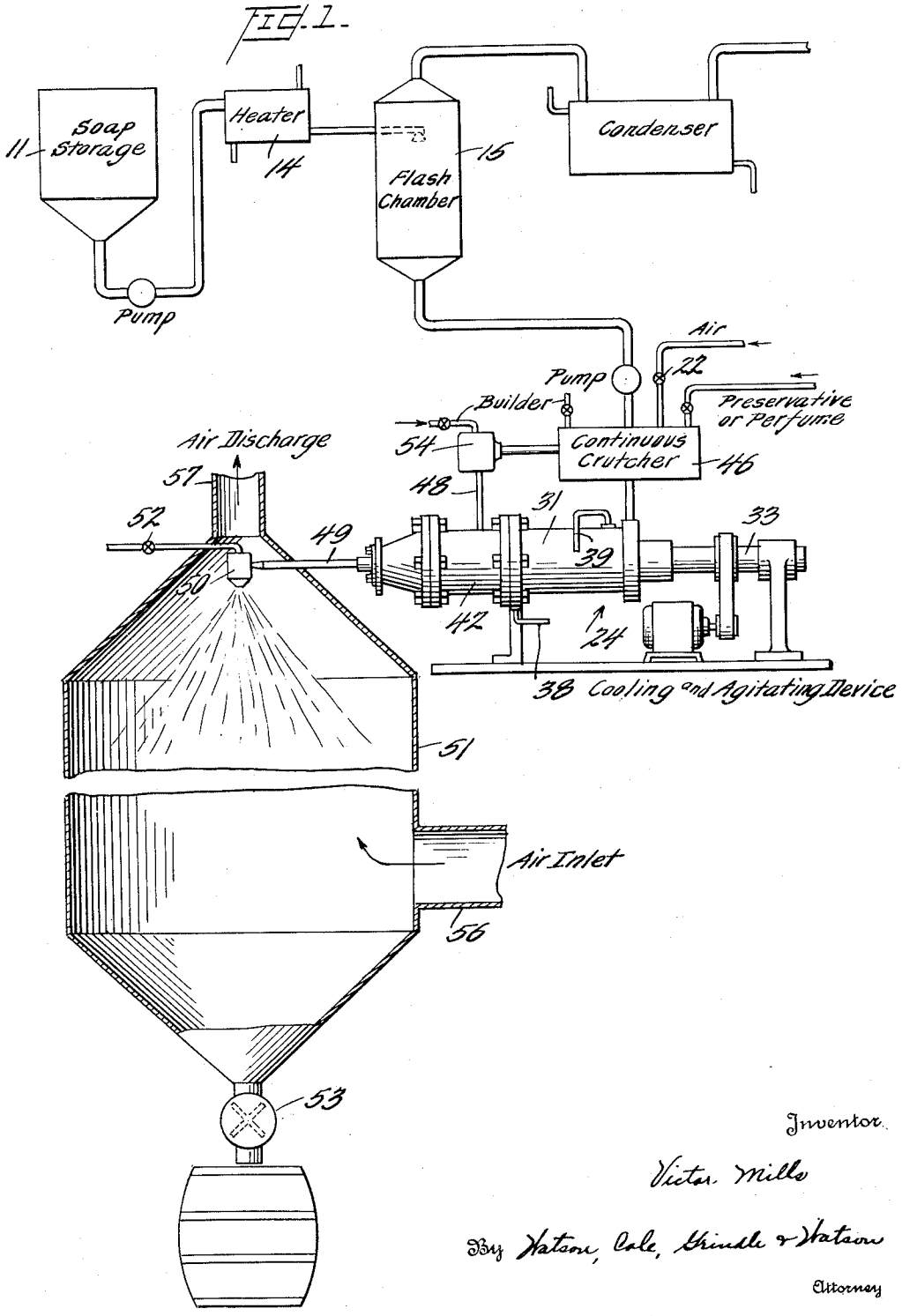
Sept. 15, 1942. V. MILLS 2,295,595

PARTICLE SOAP OF NOVEL PHASE COMPOSITION AND PROCESS FOR MAKING THE SAME

Filed June 12, 1942 2 Sheets-Sheet 1

FIG. 1.



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PARTICLE SOAP OF NOVEL PHASE COMPOSITION AND PROCESS FOR MAKING THE SAME

Filed June 12, 1942 2 Sheets-Sheet 2

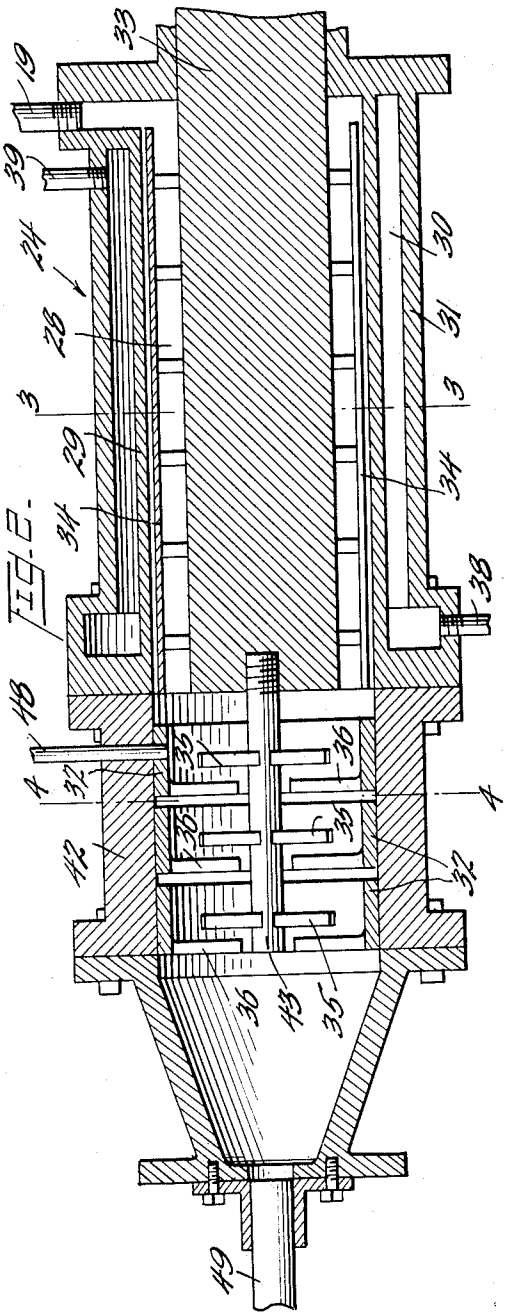


FIG. 2.

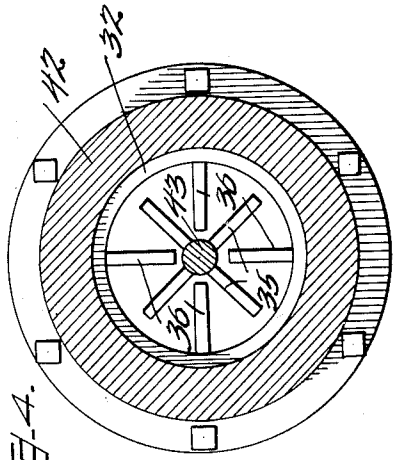


FIG. 4.

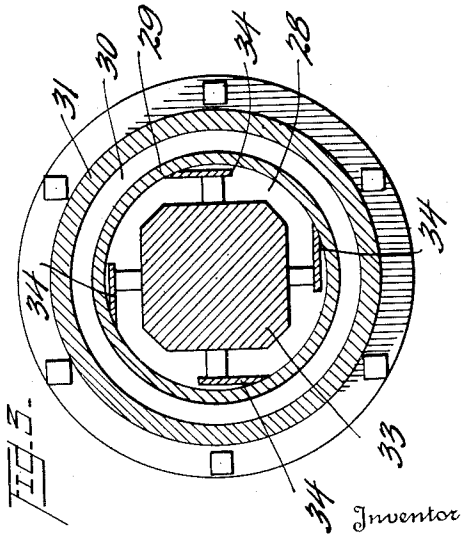


FIG. 3.

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Patented Sept. 15, 1942 2,295,595

UNITED STATES PATENT OFFICE

2,295,595

PARTICLE SOAP OF NOVEL PHASE COMPOSITION AND PROCESS FOR MAKING THE SAME

Victor Mills, Ivorydale, Ohio, assignor to The Procter and Gamble Company, Ivorydale, Ohio, a corporation of Ohio

Application June 12, 1942, Serial No. 446,757

16 Claims. (Cl. 252—368)

This invention relates to soap products in small particle form having a novel phase composition, with resulting improved properties, and to a process for making these products.

Powdered and granulated soaps for laundry and household use, both of which forms of soap will, for the sake of brevity in this specification, hereafter be included within the meaning of the terms "particle soap," or "soap in particle form," have been made in many forms and by many processes. One of the earliest and simplest methods consisted in grinding soap flakes into small particles or powder by means of a hammer mill. Later, spray drying methods were developed for making granulated soaps, which may be made either dense and relatively unpuffed, or light and highly puffed.

A high solubility rate is a much sought for property in a particle soap product, especially when combined with other desired characteristics such as free-flowing, non-balling, and non-dusting properties. Methods have been described for attaining this group of desirable qualities by careful regulation of the physical shape and structure of the individual loose particles or granules of which the product is composed. In other cases a high solubility rate has been attained by employing a soap formula containing a substantial amount of potash soap and/or coconut oil soap.

All of the aforementioned methods of making soaps in particle form, and the variations of these and other heretofore known methods with which I am familiar, produce products in which the soap is mostly, if not entirely, in the omega phase, which is a slowly soluble solid soap phase. In any of them which have contained soap in the much more rapidly soluble beta phase, this phase has been present only to the limited extent possible as a result of its spontaneous formation in the absence of mechanical agitation under the conditions prevailing when the soap cooled from fluid to solid condition.

A principal object of my invention is to produce particle soap products in which the soap is predominantly, and in some cases substantially entirely, in the more rapidly soluble beta phase.

Another object is to make particle soap products having increased solution rates because of increased beta soap content as compared with particle soap products of like chemical formula and comparable fineness produced by previously known methods.

Another object is to make particle soap products having increased solution rates because of a novel loosely knit structure, that may be produced within the individual particles or granules by the process of my invention, this structure being one which is readily penetrated and disintegrated by water.

Another object is to provide a method for controlling the beta soap content of soaps in particle form.

Other objects will appear from the following description of my invention.

In its bare essentials the process of my invention comprises transforming soap substantially, and in some cases wholly, to the beta phase by mechanical action, usually after having reduced the moisture content of the soap base substantially below 30 per cent, and then reducing the transformed soap to finely subdivided form under conditions such that its beta phase content does not revert to a significant extent to the less rapidly soluble omega phase, the processing conditions prevailing during the phase transformation step being so chosen as to favor the production of a loosely knit structure, readily penetrated and disintegrated by water, and to facilitate the subsequent subdividing step, as is hereinafter explained. During or following the subdividing step, the moisture content of the product may be further reduced if desired, taking care that the conditions prevailing during this final drying are such as to avoid reversion of the beta phase. Soap builders may be incorporated before, during, or after these processing steps if desired.

One form of apparatus suitable for the practice of the invention is shown in the accompanying drawings, in which,

Figure 1 is a diagrammatic showing of a preferred form of apparatus for use in the practice of the instant process, including the steps of reducing the moisture content of kettle soap while still in the molten condition, cooling and agitating the same, adding soap builder and further agitating the mass, and finally atomizing the resulting mixture into a cooling and drying tower from the bottom of which the finished product is removed;

Figure 2 is a longitudinal sectional view of the cooling and agitating device shown in Figure 1;

Figure 3 shows a transverse section, taken substantially on the line 3—3 of Figure 2, and

Figure 4 is a transverse sectional view on the line 4—4 of Figure 2.

The characteristics and properties of the beta and omega sodium soap phases and a method of transforming soap of suitable composition into the beta phase are described in my copending

application Serial Number 376,399, filed January 28, 1941, of which the present application is a continuation-in-part.

I have found that soap in the beta phase is formed when soap of suitable composition is mechanically agitated or worked while it is being cooled from a hot fluid state through various degrees of plasticity such that its final temperature while being agitated is reduced below a critical temperature which varies with the chemical composition of the real soap portion of the mass and with the moisture content.

The above mentioned critical temperature may be considered as the highest temperature for a given soap at which the amount of beta soap that may be produced therein by thorough agitation, followed by prompt cooling to room temperature to stabilize the phase composition resulting from this agitation, is just enough to cause a sample of the soap to exhibit the characteristic 2.75 Å. ring of beta soap when the X-ray diffraction technique described in the aforementioned application is employed. Thorough agitation of the soap at temperatures higher than the critical temperature, followed by prompt cooling without agitation, does not produce in the soap a detectable amount of the beta phase.

I determine the critical temperature by noting the value at which the characteristic beta ring appears in X-ray diffraction photographs of soap samples representing different final temperatures of cooling with simultaneous agitation in the phase transforming step of my process. A more readily available means of approximating the critical temperature is to compare sudsing rates of soap samples representing different final temperatures of cooling with simultaneous vigorous agitation with the sudsing rate of a similar sample of the soap rapidly cooled from molten to solid condition in the absence of mechanical agitation. In applying this latter test (which is merely indicative, and does not conclusively show the presence of the beta phase) it is, of course, necessary to have the samples to be compared in the same general form, preferably all in solid bar form.

The critical temperature is not the same for all soaps; it varies with changes in the "real soap formula" (by which term I mean the composition of the true soap portion of the product) and also with changes in the moisture content. For sodium soaps prepared by reducing the moisture content of kettle soap to 26 per cent, for example, the true critical temperature is found, by thoroughly agitating soap under controlled temperature conditions, to be about 160° F. when the real soap formula is composed of 20 per cent coconut oil soaps and 80 per cent tallow soaps, and it is about 150° F. when the real soap formula is composed of 50 per cent coconut oil soaps and 50 per cent tallow soaps. For the latter real soap formula the critical temperature is about 160° F. when the moisture content is 21 per cent. These critical values vary somewhat with changes in the characteristics of the fats employed. In general, for soap of a given "real soap formula" the critical temperature increases as the moisture content decreases.

Soap in the beta phase is not stable at high temperatures; under such conditions beta phase soap tends to revert spontaneously to the omega phase, or to a non-solid phase, and at the same time the solution rate of the soap, when brought back to its original temperature, decreases. Some of the beta phase soap in soaps of the aforementioned real soap formulas, containing about 20 per cent moisture and made by my new process, slowly reverts to omega phase (or reverts to a non-solid phase, and resolidifies as omega phase) at temperatures above about 160° F., and reverts more rapidly as the temperature is increased. Soaps of higher moisture content may revert somewhat even below 160° F. If heated to temperatures above 200° F. the reversion rate of 20 per cent moisture soap is so rapid that substantially all the beta phase reverts to omega or to a non-solid phase within a few minutes. Under these extreme conditions agitating action does not transform soap to the beta phase. In making powdered or granulated soap by my process it is therefore desirable to avoid subjecting the soap, after it has been cooled with simultaneous agitation, to temperatures high enough to cause substantial reversion of the beta phase, if a product of the highest obtainable solution rate is desired.

In order to facilitate an understanding of the invention and of one method whereby the invention may be practiced, apparatus which may be employed for the purpose is illustrated in the drawings, and specific language is used to describe the same. It will nevertheless be understood that no limitation of the scope of the invention is thereby intended, the practice of the invention with other forms of apparatus, capable of accomplishing the desired purpose, being contemplated.

Figure 1 shows diagrammatically the various component parts of an apparatus which may be so operated and controlled as to produce the soap of the present invention. This includes a soap storage tank **11**, a heater **14**, a flash chamber **15**, a continuous crutcher **46**, a cooling and agitating device **24**, and a spray tower **51**. It will be appreciated that the several units of the system are so shown in the drawings as to facilitate an understanding of the nature of the equipment and that the relative size of these units as so illustrated is therefore of no significance.

The continuous crutcher indicated generally at **46**, which form no part of my invention, may be any suitable ingredient proportioning and mixing means such as the devices described in Robert V. Burt's Patents 2,024,425 and 2,203,980. It will be understood that any other means of supplying molten soap to the cooling and agitating device, and of proportioning and admixing any desired additional ingredients, such as perfume, preservative, builder, air, etc., will serve my purpose.

Turning now to Figures 2, 3 and 4, constituting sectional views of the cooling and agitating device **24**, it will be noted that the soap is delivered through conduit **19** into an annular chamber **28** defined by a generally cylindrical drum **29** and a shaft **33**, the latter being supported for rotation on the axis of the drum. The drum **29** is formed of heat conducting material, such as metal, and is surrounded by a second cylindrical wall **31** to provide a cooling jacket, a cooling medium being introduced into the annular space **30** surrounding the drum **29** through an inlet port **38**, and being withdrawn therefrom through an outlet port **39**.

As is apparent from the drawings, the soap is forced to flow through the chamber **28** in a relatively thin annular layer; excellent results have been obtained in practice by the employment of a shaft **33** and a drum **29** of such dimensions that the thickness of this layer is of the order of one inch or less. A plurality of scrapers **34**, arranged as shown in Figures 3 at intervals about the shaft

33, are secured to the latter and positioned to engage and scrape the inner surface of the drum 29.

Communicating with the rear end of the drum 29, and preferably affording a continuation thereof, is a generally cylindrical drum 42.

Rigidly mounted within the drum 42 are a plurality of annular elements 32, each of which is provided with a plurality of substantially radial, inwardly directed arms 36, serving as baffles. A shaft 43, forming a reduced extension of the shaft 33, is threaded or otherwise secured to the rear end of the latter and is provided with a plurality of generally radial arms 35, disposed in proximity to the baffle arms 36 and cooperating therewith to mix the soap and to disperse throughout the mass any added ingredient which may be introduced through pipe 48. From the drum 42, the soap passes through a tapered discharge housing and is delivered to pipe 49 at the rear thereof. A motor or other suitable driving means may be employed to rotate the shaft 33 and 43, as shown in Figure 1.

The details of the cooling and agitating device disclosed herein form per se no part of the present invention. Devices of this character are disclosed, for example, in the patent to Vogt, No. 1,783,864, dated December 2, 1930, and its Reissue No. 21,406, dated March 19, 1940, and are merely representative of apparatus which are found specifically suitable in the practice of the instant invention.

The cooling device 24 is provided with an outlet pipe 49 through which the treated soap mass flows to atomizing nozzle 50 mounted near the top of the spray tower 51. Compressed air, which enters nozzle 50 through control valve 52, aids in the atomization of the pasty soap. As the finely divided soap particles fall to the bottom of the tower they are cooled, preferably to about room temperature, and are somewhat dried by an ascending column of air which may be forced in at 56 and discharged at 57. The finished product, in particle form, leaves the tower through airlock valve 53.

The spray tower indicated at 51, which forms no part of my invention, may be any suitable soap spray drying tower, such as the type described in Hall Patent 1,985,987. The atomizing nozzle 50 may be of the type illustrated in Ingram Patent 1,472,473.

The operation of the apparatus illustrated herein in carrying out my process will be understood from the following descriptive example.

Sodium soap, in the neat phase, prepared from a mixture of 15 per cent coconut oil and 85 per cent tallow by the usual well known kettle process, and containing about 30 per cent of moisture, is stored in tank 11, Figure 1. It is heated to about 390° F., under a gauge pressure of 250 pounds per square inch or more to prevent volatilization of moisture, by pumping through a closed type of heater 14, heated by high pressure steam or other suitable medium. The soap is then discharged through a spray nozzle into chamber 15, where it flashes to atmospheric pressure with volatilization of sufficient moisture to reduce its moisture content to about 23 per cent; at the same time its temperature is reduced to that corresponding to the boiling point of soap containing this reduced percentage of moisture, or about 221° F.

This hot molten soap of about 23 per cent moisture content, still in the neat phase, is then transferred by pump or other suitable means into and through the rest of the apparatus under just sufficient pressure to accomplish this transfer at the rate desired. In the cooling and agitating device, while in a thin layer not substantially greater than one inch, the soap is cooled to an average temperature of about 122° F. at which temperature it has a soft pasty consistency and is well below its critical temperature. Simultaneously air is introduced into the molten soap stream through valve 22, at a controlled rate proportionate to the rate of flow of soap, and in amount sufficient to render the mixture less viscous and therefore more readily atomized when it reaches the spray nozzle. About 20 per cent air by volume is a favorable amount. The film of soap which is chilled on the inner surface of the cooling jacket is quickly scraped off by the scrapers 34 and mixed with uncooled soap, with agitation. In the apparatus described, I find a cooling medium consisting of water between 35° F. and 65° F. to be satisfactory depending on rate of water flow and finished product properties desired, but cold brine or liquid ammonia, or other cooling medium may be used instead.

The soap then passes into and through drum 42 into which aqueous sodium silicate solution at a temperature of about 160° F. is introduced through pipe 48, at a controlled rate proportionate to the rate of flow of soap, for example by the use of a metering pump 54 driven in timed relation with the continuous crutcher 46. About 13 pounds of silicate of about 46° Bé. and having a ratio of SiO_2/Na_2O of about 3.0, per 100 pounds of 23 per cent moisture soap may suitably be used. In drum 42 the cooled and agitated soap mass is thoroughly mixed with the added silicate solution by the rotating arms or paddles 35 and by passing between the stationary baffle arms 36. The resulting mixture, which is at a temperature well below that at which reversion of phase occurs, then passes through pipe 49 to the atomizing nozzle 50 mounted in the upper part of spray tower 51.

Under the influence of a blast of air admitted into nozzle 50 through valve 52 the warm pasty soap mass is sprayed in finely divided form from this nozzle into tower 51, where it meets and falls through an ascending column of air. The volume and temperature of this air is so regulated, in well known manner, that the product does not at any time exceed about 160° F. in temperature, and that it reaches the product outlet with a moisture content of about 9 per cent and a temperature sufficiently low to avoid lumping in the product receiver or container.

The real soap portion of the particle soap thus made is about 80 per cent in the beta phase (this being judged by comparing the intensities of the characteristic beta diffraction rings with those of a soap completely converted to the beta phase by adequate working under most favorable beta forming conditions) as compared with about 20 per cent in soap comparable in composition and in processing except for omission of the step of cooling with simultaneous agitation. The soap, thus made by my process, appears to have what I term throughout this specification a loosely knit structure. In any event it is a structure which is readily penetrated and disintegrated by water. The product of the foregoing example dissolves in water in 50 to 60 seconds in a standardized solution test, whereas the similar soap made without the cooling-with-agita-

tion step required 90 to 100 seconds to dissolve when compared by the same test.

Instead of spraying the pasty soap into a current of air at atmospheric pressure, as in the foregoing specific example, it may be sprayed into a tower maintained at a pressure below that of the vapor pressure of the water in the soap. Under these conditions a sufficient amount of moisture volatilizes from the soap to lower its temperature to that at which the vapor pressure of its residual water content substantially equals the absolute pressure maintained in the tower, and a particle soap product of very desirable physical form is obtained.

In another specific example of my process and product, sodium soap of tallow mixed fatty acids was reduced to 25 per cent moisture content, and was cooled with simultaneous agitation from a hot molten condition to a temperature of 120° F. No soap builder was added in this case, and the pasty soap from the cooling device was sprayed into a cooling and drying tower from which it was removed in dry powdered form. This product contained twice as much beta soap as a comparable product made without the cooling-with-agitation step, and dissolved in water in 40 per cent less time than the latter product.

I find that a cooling device such as I have previously described, having a chilling chamber six inches in internal diameter and eighteen inches long, with free space for soap about one inch in thickness, with a shaft rotating at about 200 R. P. M., and with the jacket supplied with an adequate amount of cooling water at about 35° F., will satisfactorily cool about 400 to 700 pounds of soap per hour and transform at least two thirds of the soap to the beta or more rapidly soluble phase, when soap of about 20 to 25 per cent moisture content, and having a real soap formula consisting of 85 per cent sodium soaps of tallow mixed fatty acids and 15 per cent sodium soaps of coconut oil mixed fatty acids, is supplied to the cooling and agitating device at a temperature of about 220° F. to 223° F. and is removed from the cooling chamber thereof at a temperature of about 120° F. to 130° F. Under these conditions agitation for as short a period as one minute or even less is effective in producing a suitable amount of transformation to the beta phase.

The lower temperature limit for removal of the soap from the cooling chamber of the cooling device is just above that at which the soap loses its pasty cohesiveness and becomes stiff and resistant to flow. It is believed that this is the temperature at which the last non-solid soap in the mass solidifies. This lower limit depends upon the composition of the mass, and upon the character of the subsequent processing. In the foregoing examples the soaps would not ordinarily be cooled below about 115° F. to 120° F. because at lower temperatures they become increasingly stiff and resistant to flow.

Thus the extreme temperature range for the agitating step of my process may be defined as to its upper limit as the critical temperature of the soap mass, above which beta soap is not formed by agitating action, and as to its lower limit as just above that temperature at which the mass loses its pasty cohesiveness and becomes stiff and resistant to flow. The preferred temperature range for my process, and the only range which is operable when ordinary soap spray nozzles are employed for atomizing the soap and when the use of special high pressure pumps in the continuous crutcher is undesirable, consists of the lower portion only of the aforementioned extreme temperature range. In the upper portion of the extreme range the soap coming from the chilling and agitating device is relatively stiff and is thus unsuitable for atomizing by ordinary methods. In the lower portion of the extreme temperature range the agitated soap is, strangely, much softer in consistency, flows through pipes with relatively little resistance, and is easily atomized. Masses of soap in this preferred range will flow under the influence of gravity. I have also found that this temperature range of most favorable consistency for atomizing approximately coincides with the temperature range in which the highest proportion of beta phase may be formed by agitating action, and also with the range which produces a product of most loosely knit structure, readily penetrated by water, when the pressure to which the soap is subjected during and subsequent to its agitation does not exceed about 50 pounds per square inch (as is the case in the foregoing examples of my process). Hence my preferred temperature range for terminating the agitation just prior to subdividing the soap in finely divided form is novel, is of practical significance from the operating standpoint, and is also of significance in making a very rapidly soluble product due both to a very high beta phase content and to a loosely knit structure.

As an aid in controlling these variables, especially consistency and beta phase content, a thermometer or thermocouple may be installed in the cooling and agitating device to record the temperature of the soap passing from the cooling chamber into drum 42.

I find that generally speaking my process is applicable to, and my product may be produced from, soap of any composition suitable for detergent soap in powdered or granular form which has a real soap formula containing at least about 15 per cent of sodium soaps of saturated fatty acids having at least 16 and not more than 22 carbon atoms per molecule. The essential criterion as to operable compositions, is the capability of the soap mass to develop the beta phase upon agitation below a critical temperature. I find that sodium soaps of lauric acid and of other saturated fatty acids having a smaller number of carbon atoms than lauric acid, and sodium soaps of oleic acids and of other unsaturated fatty acids which are liquid at ordinary temperatures, when treated by themselves are not transformed into the beta phase under the conditions described herein, although mixtures of soaps of these fatty acids with sodium soaps of saturated fatty acids containing from about sixteen to about twenty-two carbon atoms per molecule may, if the mixture contains at least fifteen per cent of such saturated sodium soaps, readily be transformed to the beta phase. When a product containing a very high beta phase content is desired the real soap formula may well contain at least about forty per cent of such saturated sodium soaps.

Sodium soaps made from soap making fats substantially equivalent to tallow, such as palm oil and hydrogenated vegetable and marine oils, may be substituted in whole or in part for tallow in preparing the soap for use in my process. Likewise the soap for my process may contain substantial proportions of sodium soaps of coconut oil or of other tropical nut oils of the coconut oil type such as palm kernel oil, babassu oil,

and cohune oil, although satisfactory products have been made by my process without the use of any oils of this type. Soap making fats having characteristics different from those of tallow and coconut oil may also be used; likewise water soluble salts of potash or other basic materials, such as nitrogen bases, may be substituted in part for sodium soaps. By analogy with the operating conditions described for the examples given herein, those skilled in the art will readily recognize suitable operating conditions for any given formula.

My process is applicable in the manufacture of substantially unbuilt soaps in particle form as well as soaps containing varying amounts of suitable soap builders. Builders incorporated in liquid form may be introduced prior to the step of cooling the soap with simultaneous agitation, provided the amount of builder thus incorporated is not so excessive in amount as to interfere with the formation of the beta phase. Alternatively, liquid builders may be incorporated immediately after the step of cooling the soap with simultaneous agitation, as in the preceding example, or they may be introduced into the soap at the atomizing nozzle in the spray tower. Builders in dry solid form, such as soda ash and powdered sodium phosphate, may be mixed with the cooled dry particle soap after it discharges from the bottom of the spray tower. Suitable builders include sodium silicate, sodium metasilicate, sodium carbonate, trisodium phosphate, sodium pyrophosphate, sodium perborate, and in general all builders that are appropriately used in any particle soap product.

Aeration of the soap prior to atomizing, and preferably prior to the step of cooling with simultaneous agitation, is desirable as an aid to atomization, although such aeration is not essential.

The most favorable moisture range for the soap when subjected to the cooling and agitating step is from about 10 per cent to about 27 per cent.

The moisture content of the final product of my process may be controlled in accordance with well known practice in making particle soaps of conventional types, although precautions should be taken to avoid heating the soap to a temperature high enough to cause reversion of phase. The moisture content of the product is usually between about 5 per cent and about 20 per cent, although in some cases it may be slightly lower than 5 per cent or as high as about 22 per cent.

The preparation of soap of substantial beta phase content in flake form is disclosed and claimed more especially in my copending application, Serial No. 446,758, filed concurrently herewith.

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

1. A soap product in particle form, of formula suitable for household and laundry use, containing a substantial proportion of sodium soap in the beta phase, and having a structure typical of a soap which has been whipped mechanically while at a temperature such that the mass is in a condition of pasty cohensiveness and while within the range in which beta soap is formed on agitation, and which has been maintained, both during said whipping and thereafter until subdivided in particle form, at pressures sufficiently low to avoid substantial compacting of the soap such as would substantially destroy its loosely knit structure, which is readily penetrated and disintegrated by water.

2. A detergent soap in particle form having a real soap formula containing at least about fifteen per cent of sodium soaps of saturated fatty acids having not less than sixteen nor more than twenty-two carbon atoms per molecule, and having a structure and phase composition characteristic of a soap which has been effectively agitated and reduced to subdivided form while above a temperature at which the soap loses its pasty cohesiveness, and within the temperature range in which the beta phase as shown by X-ray diffraction photograph is formed in substantial amount upon agitating, and which has subsequently been cooled substantially to room temperature, said soap in particle form containing a substantial proportion of soap in the beta phase uniformly distributed therethrough.

3. The product of claim 2, in which said soap in particle form comprises as its principal detergent ingredient sodium soaps predominantly in the beta phase.

4. The product of claim 2, in which said soap in particle form comprises as its principal detergent ingredient sodium soaps at least two thirds of which are in the beta phase.

5. The product of claim 2, in which said soap in particle form is a spray processed soap containing as its principal detergent ingredient sodium soap predominantly in beta phase.

6. The product of claim 2, comprising a substantial amount of a soap builder as well as said soap in the beta phase.

7. The product of claim 2 in which said real soap formula is substantially equivalent to soaps of tallow fatty acids.

8. A soap product in particle form, of formula suitable for household and laundry use, containing a substantial proportion of sodium soap in the beta phase, and having a structure typical of a soap which has been whipped mechanically while within the range in which beta soap is formed on agitation, and while within the more limited range defined as to its upper limit as the highest temperature (below the critical temperature of the mass) at which the mass will flow under the influence of gravity and defined as to its lower limit as the lowest temperature at which the mass possesses a pasty cohesiveness and is not stiff and resistant to flow, and which has been maintained, both during said whipping and thereafter until subdivided in particle form, at pressures sufficiently low to avoid substantial compacting of the soap.

9. In a process of manufacturing detergent soap in particle form containing a substantial amount of soap in the beta phase, the steps which comprise effectively agitating a soap mass while said mass is within a temperature range in which the beta phase of said soap is formed in substantial amount upon agitating and while above that temperature below which said mass loses its pasty cohesiveness, terminating this agitating step while within said temperature range and while above that temperature below which said mass loses its pasty cohesiveness, and reducing said agitated mass to finely subdivided form, maintaining said soap during the steps of its manufacture subsequent to said agitating step at temperatures below those at which substantial reversion of the beta phase of said soap to another phase takes place.

10. The process of claim 9 in which the soap of

said soap mass contains at least fifteen per cent of sodium soaps of saturated fatty acids containing from about sixteen to about twenty-two carbon atoms per molecule and in which said mass has a moisture content between about ten per cent and about twenty-seven per cent.

11. A process for preparing soap in particle form comprising soaps predominantly in the beta phase which comprises the steps of preparing a mass of molten soap containing from about ten per cent to about twenty-seven per cent moisture, the real soap formula of which contains at least about fifteen per cent of sodium soaps of saturated fatty acids containing not less than sixteen nor more than twenty-two carbon atoms per molecule, cooling said soap mass to a temperature between about 160° F. and 115° F., within which range beta phase soap is formed on agitation and said mass is in a condition of pasty cohesiveness, effectively agitating said soap while establishing and maintaining same within said temperature range to transform a substantial portion of the soap to the beta phase, reducing said cooled and agitated soap to finely subdivided form by atomization, and further cooling and drying said atomized soap by spray drying without rise of temperature above 160° F.

12. The process described in claim 11, in which air is introduced into and dispersed throughout the soap prior to its atomization, in amount sufficient to render it more readily atomizable.

13. The process described in claim 11, in which a soap builder is introduced in controlled proportion into the soap prior to said atomization.

14. The process described in claim 11, in which said sodium soap are soaps of fatty acids of the tallow type.

15. The process of claim 11, in which the real soap formula of the mass contains at least about forty per cent of sodium soaps of saturated fatty acids containing not less than sixteen nor more than twenty-two carbon atoms per molecule, and in which the particle soap product contains soap predominantly in the beta phase.

16. In a process of manufacturing detergent soap in particle form containing as its principal detergent ingredient sodium soaps predominantly in the beta phase, the steps which comprise effectively agitating a soap mass while said mass is within a temperature range in which the beta phase of said soap is formed in substantial amount upon agitating, said range being further limited as to its upper limit as the highest temperature (below the critical temperature of the mass) at which the mass will flow under the influence of gravity and as to its lower limit as the lowest temperature at which the mass possesses a pasty cohesiveness and is not stiff and resistant to flow; subdividing said mass into finely divided form while within said temperature range; and partially drying and cooling the subdivided particles to form said soap product.

VICTOR MILLS.