Dec. 11, 1951 V. MILLS 2,578,366

CONTINUOUS PROCESS FOR NEUTRALIZING FATTY ACIDS

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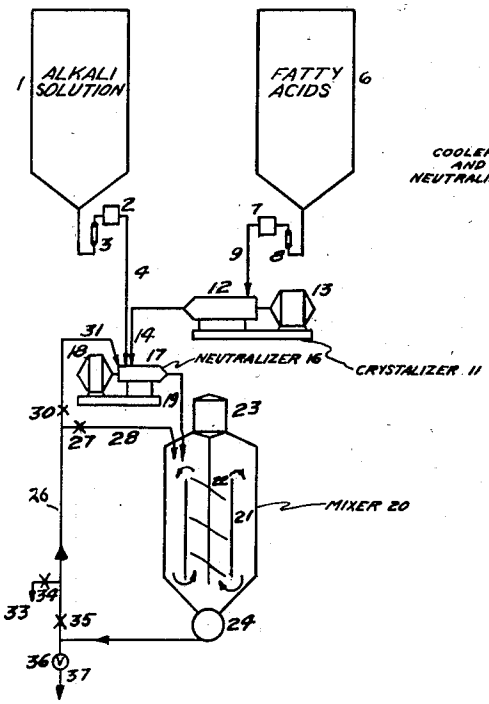


FIG. 1

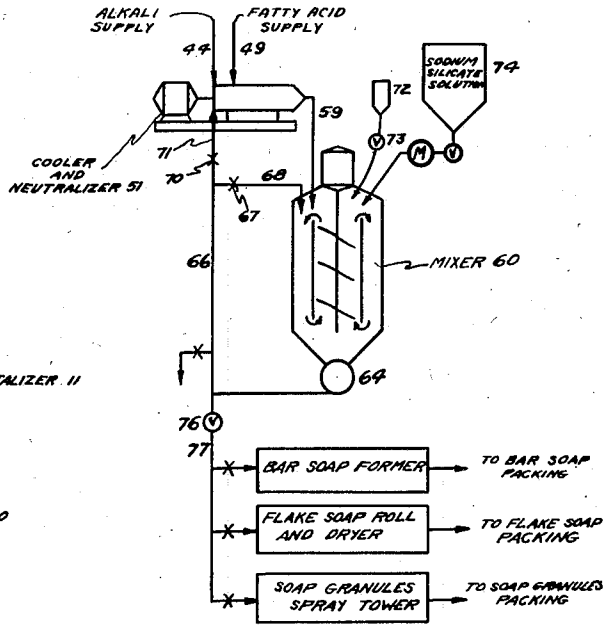


FIG. 2

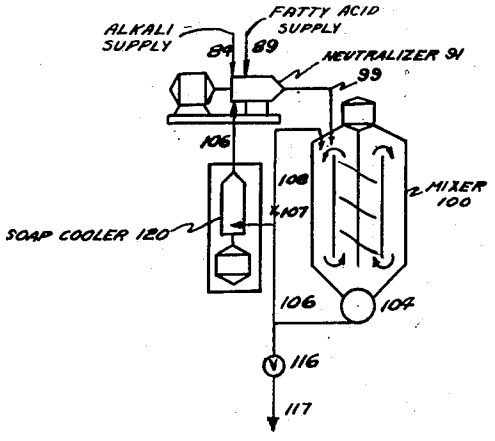


FIG. 3

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CONTINUOUS PROCESS FOR NEUTRALIZING FATTY ACIDS

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This invention relates to the manufacture of water-soluble soaps from fatty acids and alkaline materials. More particularly, it relates to a process of neutralizing soapmaking fatty acids to produce directly, without a drying step, soap having a moisture content substantially below that of kettle soap.

Hitherto it has been thought necessary, when continuously reacting flowing streams of fatty acids and alkali and producing soap which is molten when formed, either to use a relatively dilute aqueous solution of alkali or to have the fatty acids exceedingly hot. In each case the object sought for was to provide conditions—either high dilution or high temperature—such that the resulting soap was relatively fluid. It has been generally considered impractical or impossible to obtain satisfactory results, especially as to soap purity, when the soap formed was so viscous as to impede complete mixing of the reactants, or so viscous as to aggravate the danger of local overheating from failure to dissipate the large amount of heat that is liberated by the reaction. The prior practices in which the soap has been made quite fluid by relatively high water content or by high temperature have, however, serious objections which my process overcomes.

When the first of these prior procedures is employed, i. e., when the fatty acids are reacted with a fairly dilute solution of alkali, the resulting soap frequently has a moisture content in the neighborhood of thirty per cent to thirty-five per cent, which is higher than is desirable in most commercial forms, and consequently it is necessary to subject it to a subsequent drying operation—a step which it is one of my objects to avoid.

When the second of these procedures is employed, i. e., when the alkali is caused to react with the fatty acids at very high temperature, frequently at about 250° F. or higher and well above the temperature at which the soap boils at atmospheric pressure, high pressure apparatus is usually required, and the quality of the product tends to be impaired. The avoidance of expensive high pressure equipment and the avoidance of darkening of the soap, and of other adverse quality effects which result from exposure of fatty materials to high temperatures, are objects of my invention.

It has been proposed in the past to combine the cooling and drying of the hot dilute soap that results from such procedures into one operation known as "flash drying," but this too is unnecessary in the present process.

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A primary object of my invention is to provide conditions, in the continuous neutralization of soapmaking fatty acids with relatively concentrated alkali solutions, such that injurious overheating—including local overheating—is avoided. Another object is to provide conditions, in this process, such that the soap produced is uniform in purity—i. e., such that it is substantially and uniformly free from unreacted fatty acids and/or alkali and from variations in color and color stability.

Another object of my invention is to produce soap which is, when so desired, predominantly in the beta phase—this being a solid crystalline soap phase of ready solubility—directly from fatty acids and an aqueous solution of alkali. An alternative object is to produce soap, from the same starting materials, in the less readily soluble omega phase, when this solid crystalline form of soap is preferred. Further objects are to make soap of both a desired moisture content and a desired phase composition in a single continuous operation, and to accomplish this without employing a drying operation of any kind.

The present invention is based upon my discovery that it is possible to react fatty acids with relatively concentrated aqueous solutions of alkali—such as to produce soap containing substantially less moisture than kettle soap (which normally contains about 30 per cent) and preferably containing from about 12 to about 27 per cent of moisture—without the employment of the very high temperatures which have been used in the past, when certain precautions are taken to avoid local overheating and to permit effective stirring and mixing of the reaction mass. In this process, local overheating is avoided both by the choice of relatively low and specifically chosen ranges of temperatures, in which the soap is in what may be called a "pumpable condition," and also by providing a heat absorbing medium in close proximity to the point or points where the neutralization reaction occurs.

One advantageous manner of providing such a heat absorbing medium is by precooling the fatty acids to a point such that a substantial proportion of them are in the form of solidified crystals suspended as a slurry in a mother liquor of unsolidified fatty acids. When fatty acids and alkali are caused to react under these conditions, a substantial proportion of the heat of reaction is used in melting the fatty acid crystals, and

even in the absence of thorough mixing or stirring there is much less tendency towards local overheating than there is when both reactants are fully liquefied to start with.

Another manner of providing a suitable heat absorbing medium is by conducting the neutralization reaction within an efficient continuous cooling device, such as the cooling and agitating device illustrated in Mills Patent 2,295,594. To avoid corrosion difficulties I prefer to use a cooling device made of stainless steel, or other metal which is resistant to attack by fatty acids and by caustic. When fatty acids and alkali are caused to react in such a device it is possible to absorb the heat of reaction almost as fast as it is created.

Another advantageous manner of providing an effective heat absorbing medium is by recirculating through the mixing zone, where the fatty acids and alkali solution meet, a large quantity of the fluid soap previously made by the neutralization reaction, the soap thus recirculated desirably being cooled somewhat before it is brought back to the mixing zone. Recirculation of previously made soap through the mixing zone is highly advantageous for other reasons, besides its function of absorbing heat of the reaction. It serves to dilute or disperse the reactants in a manner favorable to rapid and even progress of the reaction, and also to smooth out such irregularities as there may be in the proportioning of the fatty acids and the saponifying agent. In fact, the benefits derived from this recirculation of soap through the mixing zone are so great that I prefer to follow this practice in most cases, and even to combine it with another means of absorbing heat of reaction, such as one of those referred to in the two preceding paragraphs. It is frequently advantageous to merge one of the proportioned streams of reactants (preferably the stream of fatty acids) with the recirculated stream of soap ahead of the point at which the proportioned stream of the other reactant (preferably the alkali solution) enters the process and at which the intimate admixture of the two reactants occurs.

In speaking of the condition of soap due to its temperature, I have used the expression "pumpable condition." By this I mean that the soap is of a consistency such that it may be forced to flow through factory pipe lines of moderate diameter (e, g. 4 inch to 6 inch) by means of a suitable pump, without the employment of excessively high or impractical pressures. When soap is in such a condition it may be stirred, preferably by means of a fairly powerful agitator, so effectively that all portions of the mass may be admixed, whereby complete neutralization is brought about and a uniform product is produced.

Hydrated soap within the moisture range of my process is in a pumpable condition over one or more critical ranges of temperature, these ranges for any particular soap depending to a large extent upon its moisture content and to some extent also upon its chemical composition—including within the meaning of this term the alkali employed in making it (whether potash or soda or ammonia or one of the hydroxy amines, for example), the molecular weight and degree of unsaturation of the fatty acids employed, and the amount and kind of associated materials such as glycerin, salt, free alkali, sodium silicate, sodium carbonate, sodium phosphate, etc.

I have found that in the case of neat soaps of the compositions normally employed as detergents in household and industrial use, suitable temperatures for effectively stirring the soap to complete the mixing of the fatty acids and alkali and to produce a uniform product, and for pumping the soap, are most likely to be found some distance below, and/or well above, the temperature point at which the first solid soap crystals appear in the neat soap as it is cooled (in the absence of supercooling), or the temperature at which the last solid soap crystals melt to neat soap when the soap is warmed. This point, which may be called the "complete melting point" of the soap, may, if desired, be accurately determined for any given soap composition by dilatometer measurements supplemented by X-ray diffraction photographs, as discussed by Ferguson, Rosevear, and Stillman in Industrial and Engineering Chemistry, September 1943, pages 1003–1012. For most practical purposes, however, the determination by such laboratory methods is entirely unnecessary, and simple observation of the behavior of the soap under practical operating conditions may be relied upon. Such practical observation is to be preferred to more scientific tests, in fact, because the more desirable ranges for mixing the soap are sometimes shifted, as to temperature limits, depending upon the amount and type of agitation to which the soap is subjected. These desirable ranges may also be shifted upward and/or downward in temperature, when desired, by adding more or less salt to the alkali solution.

In the case of those soaps which form the beta phase as described in my U. S. Patent No. 2,295,-594, the complete melting point of the soap may be considered as coinciding with the "critical temperature" which is referred to and fully explained in that patent. Slightly above this critical temperature, soap undergoing treatment by the process of that patent is inclined to be extremely firm and gummy, as indicated in the second table on page 4 of the aforesaid patent, and in this rather narrow temperature range it is not in a desirable condition for pumping or for thorough stirring or mixing. On either side of this temperature region of quite gummy soap, i. e. both above it and below it, temperature ranges favorable for my process exist. (My use of the term "gummy" does not have reference to "middle soap" which is sometimes spoken of as gummy soap.)

Thus in my process I preferably employ temperature conditions such that the soap formed by the neutralization of the fatty acids with the alkali is either safely above the temperatures at which this gummy soap is found and is within a temperature range in which the soap is of pumpable consistency and yet is not at a damagingly high temperature, or is below the complete melting point of the soap and within the temperature range in which a mixture of neat soap and crystallized soap is found—this latter being a range in which the soap may be said to have a pasty consistency and also a characteristic pasty cohesiveness. In this lower temperature range, below the complete melting point, most soaps—as the temperature is gradually lowered—become thinner in consistency for a time, remain relatively thin for some degrees, and then rather abruptly become stiffer as the solidification of the soap becomes more complete. This low temperature range of thin consistency, which is one of the preferred temperature ranges for the present process, is described on page 4 of my U. S. Patent No. 2,295,595. The lower limit of this

temperature range may be defined as just above that temperature at which the soap loses its pasty consistency and becomes tough and resistant to flow as it approaches complete solidification.

When fully molten neat soap of ordinary composition, such as is used in household and commercial cleansing soaps, and having a moisture content within the range of my process, is heated under pressure to increasingly high temperatures—above its boiling point when measured at atmospheric pressure—its consistency does not as a rule become substantially thinner, contrary to popular belief. In many instances it may become so much stiffer as the temperature increases that pumping difficulties become acute. For this reason, and also because the higher temperatures are themselves injurious and because they necessitate the expenses of high pressure equipment and of subsequently cooling the soap, I prefer not to exceed the atmospheric boiling point of the soap mass in the temperature reached in my process. This boiling point is in the neighborhood of 220° F. to 235° F., depending upon moisture content, for most neat soaps commonly employed in the process.

For sodium soaps of the more commonly used soap-making fatty acid mixtures, within the moisture range of 19 per cent to 25 per cent, my most perferred temperature ranges for mixing the soap as it is formed and completing the neutralization of the last traces of fatty acids are from about 130° F. to about 150° F. and from about 190° F. or 195° F. to about 215° F.

For any soap composition that may be encountered, suitable temperature ranges for the mixing and stirring operations of my process will readily be recognized by those skilled in soap making, when guided by the information herein set forth. The choice between pumpable temperature ranges below and above the complete melting is largely dependent upon the phase, and the resulting soap properties, desired in the finished product.

In making soaps of composition such that the beta phase of the soap may be made by mechanically working solidified soap crystals at suitable temperatures—e. g. soaps having a real soap formula containing at least 15 per cent of sodium soaps of saturated fatty acids having 16 to 22 carbon atoms per molecule (see Patent 2,295,594)—I regulate the temperature of the reactants in my present neutralization process so that the soap as formed is in the temperature range below its complete melting point when I want the resulting soap to be substantially or predominantly in the beta phase; and, alternatively, I so regulate conditions that the soap as formed is in the temperature range above its complete melting point when I want the soap to have the properties characteristic of the omega phase. In this latter case, when soap of omega phase alone is preferred, it is desirable to conduct the subsequent processing of the soap formed by the neutralization reaction in a manner which includes fairly rapid cooling, without agitation, from the fully molten to the fully solidified condition.

In making soaps of compositions such that the beta phase is not formed by mechanically working the soap at temperatures within at least parts of the pasty range below its complete melting point—e. g. sodium soaps made entirely from coconut oil—the soap resulting from the process steps herein described and claimed will normally be in the omega phase no matter what portion of my broad temperature range is chosen for the neutralization and mixing operations.

The moisture content of the soap formed by the neutralization reaction is readily controlled by any skilled soapmaker. The amount of sodium hydroxide, or of such other alkali as may be used, which is needed to completely react with the fatty acids is calculated from the saponification value of the acids, and the amount of water created by the reaction is also calculated; and allowance is made for the moisture content of the fatty acids if this is appreciable. The additional water required to produce a soap of the desired moisture content is then calculated, and this amount of water is used in preparing the solution of the alkali.

The control of the temperature of the soap as formed may be accomplished in various ways, depending primarily upon the particular heat absorbing medium, or combination of such media, employed.

If crystallized fatty acids, slurried in liquid fatty acids, constitutes the heat absorbing medium the most important control is the degree of crystallization of this slurry—this being a measure of its total heat content. For any given temperature of the alkali solution it is a simple matter to vary the degree of crystallization of the fatty acid slurry to obtain any desired soap temperature within the attainable range. If calculations are preferred, rather than empirical control, the following approximate constants for typical mixtures of fatty acids of the commercially more important types may be used:

	Tallow Fatty Acids	Coconut Oil Fatty Acids
Heat of crystallization, B. t. u./lb.	85	85
Heat of neutralization with NaOH, B. t. u./lb. fatty acids	60	90
Specific heat of fatty acids, B. t. u./lb. at 130° F.	0.5	0.5

If the walls of a cooling chamber, such as a "Votator," constitute the heat absorbing medium, the more important controls are the temperature and rate of flow of the water, brine, or other cooling liquid, which is caused to flow through the jacket provided in the walls of this chamber. Knowing the cooling characteristics of the particular cooling device employed, it is readily possible to calculate, or quickly determine by trial, the temperature and/or rate of flow of cooling liquid for any desired soap temperature, taking into consideration the temperature and amounts of the alkali solution and of the fatty acids entering the chamber.

If a recirculated stream of previously made soap is the primary effective heat absorbing medium employed, the important controls usually are the temperature of this recirculated stream and its amount per unit of time in relation to the amount of new soap formed by the reaction—especially if the stream of soap being recirculated is subjected to a cooling operation before it enters the fatty acid-alkali mixing zone.

In the event that recirculation of soap is practiced, together with the employment of a second heat absorbing medium—e. g. fatty acid crystals or the walls of a cooling chamber—the recirculated soap stream is usually not cooled, and the temperature control is effected primarily as in the case of the second medium alone. It is obvious, however, that there are many possible ways in which temperature control may be effected in

my process, and my invention is not limited to any particular manner of control.

I will now describe several examples of my process, typifying several ways in which the invention may be practiced. In this connection reference will be made to the drawings which illustrate some of the different forms of apparatus which may suitably be employed in carrying out the process.

In the drawings, all of which are schematic, Figure 1 shows apparatus particularly useful for my process when crystallized fatty acids are employed for temperature regulating purposes; Figure 2 shows similar apparatus particularly adapted to the neutralization of fatty acids within a continuous cooling device; Figure 3 shows apparatus which may be used when a recirculated stream of previously formed and cooled soap is the only effective heat absorbing medium at the point where the reaction occurs. Further explanation of the apparatus indicated in the several drawings is to be found in the following descriptions of specific examples of my process.

Example 1.—In this example reference is made to the apparatus shown schematically in Figure 1.

Tank 1 is kept supplied with an aqueous solution of caustic soda containing 40.8% sodium hydroxide, 1% salt, and 58.2% water by weight, the temperature of the solution being kept at about 80° F. Tank 6 is kept supplied with a melted mixture of 80% tallow fatty acids and 20% coconut oil fatty acids, at any convenient non-injurious temperature.

Fatty acids are continuously withdrawn from tank 6 and forced, by pump 7, through the fatty acid crystallizer 11 and into the neutralizer 16 and the several connecting pipe lines. At the same time caustic soda solution is continuously withdrawn from tank 1 and forced, by pump 2, through the connecting pipe lines into the neutralizer 16. These streams of fatty acids and caustic solution are proportioned so as to deliver these two reactants at rates which are substantially equivalent chemically. This proportioning is accomplished, usually after determinations of the saponification value of the fatty acids and the sodium hydroxide concentration of the caustic solution, by means of any suitable proportioning device—several satisfactory forms of these being well known and commercially available; or it may be accomplished by hand control, the pumps 2 and/or 7 in this case being either variable speed pumps, or fixed speed pumps each equipped with a by-pass or "run-around" line and suitable valves to permit regulating the net delivery of the liquid being pumped. Flow meters 3 and 8 are conveniently employed to give a visual index of the rates of flow of the two reactants.

The fatty acids crystallizer may be any suitable crystallizing device, such as the enclosed horizontal continuous crystallizer shown at 11 in the drawing. This is a cooling device of the general type shown in Mills U. S. Patent No. 2,295,594 (from which the agitating zone beyond the cooling zone has been omitted), comprising a cylinder surrounded by a cooling jacket, through which cold water or brine may be passed, a shaft concentric with the axis of the cylinder and occupying most of the space within the cylinder, leaving but a narrow annular space through which the material to be cooled is passed, blades mounted on the shaft designed to scrape the entire inner wall of the cylinder as the shaft rotates, a motor to turn the shaft in the cylinder, and suitable pipe connections and valves to conduct the cooling liquid into and out of the jacket and to admit the fatty acids into the annular space at one end of the cylinder and discharge them at the other end. As described and illustrated in the Mills patent just mentioned (page 4, right, beginning at line 68, and Fig. 3) the annular space of this cooling device may be about one inch in thickness and the scraper shaft, bearing four blades, may be rotated at about 200 R. P. M., thus providing agitation and removal of chilled material from the cooling surface at a frequency of about 800 times per minute. As the stream of fatty acids passes through the crystallizer, enough heat is extracted by the indirect aid of the cooling fluid to partially solidify the fatty acids and to produce a slurry of crystallized fatty acids suspended in liquid fatty acids. A thermometer in the outlet pipe may be used as a preliminary guide for the control of the fatty acid crystal formation, and a thermometer in the soap outlet line 19 leading from neutralizer 16 is conveniently used as a final guide for the control of the cooling which is brought about in the crystallizer. In the present example, with the caustic solution delivered to the neutralizer at 80° F., a suitable amount of crystallization in the fatty acid slurry issuing from the crystallizer is about 40%.

The proportioned streams of alkali flowing through pipe 4 and of fatty acid slurry flowing through pipe 14 are brought together in neutralizer 16, this being a simple mixing device, such as a horizontal cylindrical stainless steel vessel in which there is a horizontal shaft driven by motor 18. The shaft is equipped with sturdy agitator blades, and the inner wall of the neutralizer chamber may be equipped with fixed hold-back blades, the whole device being designed to thoroughly mix and agitate the reactants and the resulting soap. The construction of the neutralizer is sufficiently sturdy and the motor sufficiently powerful to effect high speed and thorough mixing even though the material passing through the neutralizer is a viscous liquid or pasty semi-liquid. The neutralization of the fatty acids by means of alkali occurs so rapidly that the shell of the neutralizer need be no larger than about 6 inches in internal diameter and 15 inches in length for a production rate equivalent to 5000 pounds of fatty acid per hour. A rotor speed of 1700 R. P. M. has been found suitable in such a device, with clearances between the rotating blades and stationary hold-back blades of approximately ⅛ inch. The neutralizer may be either open to the atmosphere or closed. A neutralizer closed to the atmosphere and designed to be completely filled by the reaction mixture is the form I prefer, because this eliminates problems caused by spattering, cold soap crust formation, and aeration, such as arise when an open mixer is used, and because it results in flow of reactants through the neutralizer in a more positive and uniform manner.

In the present example the heat of neutralization is sufficient to melt the crystals of fatty acid in the slurry and to raise the temperature of the reaction mixture to about 130°–135° F. The soap formed, which in this case has a moisture content of approximately 23.7%, is a readily pumpable yet pasty mass at this temperature.

The soap from the neutralizer, under the impelling action of pumps 2 and 7, is forced out of pipe 19 and discharged into a large mixer 20,

which is usually open to the atmosphere. This mixer, which may well have a soap capacity of 6000 pounds for a production rate equivalent to 5000 pounds of fatty acids per hour, is designed to thoroughly mix and circulate the soap charge passing through it. To this end it may have a design similar to the conventional soap crutcher, i. e. having a centrally mounted helical agitator 22 rotating in a vertical cylinder or sleeve, with an annular space 21 surrounding this sleeve through which the soap is free to move under the impelling action of the helix. This mixer serves to complete the mixing of the reactants and the saponification of the fatty acids, to the minor extent that this may not have been completed in neutralizer 16, and to "iron out" most of such irregularities in the proportioning of fatty acids and alkali as may have resulted from uneven operation of the respective supply pumps 2 and 7 together with their accessory regulating devices.

The thoroughly saponified soap, now quite uniform in composition, is delivered from the bottom of mixer 20 by means of pump 24 into the delivery conduit 37 (valve 35 being closed), whence it is conducted to whatever further processing is desired—for example, forming into soap flakes, or soap granules, or bar soap. The soap delivered from pipe 37, in this example, is predominantly in the beta phase.

Such other materials as it may be desired to incorporate with this soap, such as sodium carbonate, sodium silicate, sodium phosphate, or any other soap builders, or preservatives, coloring matter, perfume, air, etc., may be incorporated at any convenient place in the system at the discretion of the soapmaker. It may be convenient to bring some of these materials into the system at the neutralizer 16, or into the mixer 20, or, if desired, in or beyond the delivery line 37.

An alternative and advantageous method of handling the soap withdrawn from mixer 20 is to operate pump 24 at a capacity several times greater than the soap production rate, and to divide the flow of soap coming from pump 24 so that the greater part of it is recirculated through pipes 26 and 28 and back into the upper part of the mixer, the amount withdrawn through line 37 in this case being just enough to maintain the level of soap in mixer 20 substantially constant, and usually slightly above the top of the helical agitator. When operating in this manner valves 27 and 35 are opened, valve 30 is shut, and control valve 36 is regulated so as to maintain the desired soap level in mixer 20.

Another alternative method of operation is to recirculate soap through pipes 26 and 31 into the neutralizer 16, valve 30 being open in this case and valve 27 being closed.

I find it good practice to draw samples of the soap either from the delivery line 37 or through a special sample cock 33, controlled by valve 34, at frequent and regular intervals, and to analyze these samples promptly for free fatty acid and/or free alkali. With the information thus obtained, the supply of alkali through meter 3 and/or of the supply fatty acids through meter 8 may be adjusted to obtain and to maintain substantial neutrality, or any desired degree of alkalinity or acidity in the finished soap.

Example 2.—In this example reference is made to the apparatus shown schematically in Figure 2.

A molten mixture consisting of 85% tallow fatty acids, 10% cottonseed foots fatty acids, and 5% rosin, this mixture having a saponification value of 200, is delivered at constant rate through pipe 49 to the continuous cooling and neutralizing device indicated at 51.

Simultaneously a caustic soda solution containing 31% NaOH by weight, having a temperature of 95° F., is delivered through pipe 44 to cooler and neutralizer 51 at a rate of approximately 46 pounds for every 100 pounds of fatty acids flowing through pipe 49.

In cooler and neutralizer 51, which may be constructed along the same general lines as crystallizer 11 shown in Figure 1 and referred to in Example 1, the fatty acids and caustic become intimately mixed and react together to form soap containing about 26 per cent moisture. The flow of cooling fluid passing through the jacket of the cooler and neutralizer 51 is regulated so that the soap delivered through pipe 59 will be at a temperature of 150° F. This soap under these conditions has a rather heavy consistency, but is pumpable, and has properties characteristic of soap containing substantial amounts of the beta phase.

The mixing and the saponification, and further blending to attain a high degree of uniformity, are completed in mixer 60 in the same general manner as in mixer 20 of Figure 1, referred to in Example 1. As in that example, it is usually advantageous to recirculate soap from mixer 60 through lines 66 and 68, or through lines 66 and 71. Another advantageous method of operating is to recirculate soap at a relatively large volume rate through lines 66 and 68, and to divert soap at a relatively slower volume rate from line 66 through line 71, this being accomplisher by opening both valves 67 and 70, and regulating the settings of these valves to obtain the desired relative amounts of flow through lines 68 and 71 respectively.

A mixture of perfume and preservative is fed from tank 72 through control valve 73 to mixer 60, at a rate equivalent to 0.01% of the combined delivery rates of flow through pipes 44 and 49. Simultaneously sodium silicate solution, containing 40% solids and 60% water, is delivered from tank 74 through a suitable control valve and pipe, and through a meter 75, into the top of mixer 60, the rate of feed being 10% of the combined rates of flow of materials through pipes 44 and 49 figured on a weight basis. Controllable feeding means from tanks 72 and 74 may, if desired, be interconnected with the flow control means of the fatty acid and caustic supplies in order to obtain more precise control of the proportions of these added ingredients. The perfume, preservative, and builder thus added to the soap are uniformly incorporated by the mixing action of the agitator in mixer 60 and by the recirculation through line 66 and one or both of lines 68 and 71.

The product of the process of this example, i. e. soap of about 29% moisture (3% of it introduced with the silicate, beyond the point of neutralization), at a temperature of about 160° F., may be withdrawn through control valve 76 in line 77 and delivered to a granules spray tower, such as the spray drying equipment shown in Hall U. S. Patent No. 1,985,987, where the soap is sprayed through a current of warm air to reduce its moisture content to about 15% and to cause it to solidify in finely divided granular form.

As indicated in Figure 2, the soap from the

process may alternatively be finished in other forms than granular, e. g. as bar soap or as soap flakes or chips, to name but two of the more common commercial forms.

Example 3.—In this example reference is made to the apparatus shown schematically in Figure 3. A mixture consisting of fatty acids (⅓ commercial distilled oleic acid and ⅔ fatty acids of tallow) and having a temperature maintained at about 115° F., is delivered at substantially constant rate through pipe 84 to the neutralizer indicated at 91.

At the same time an aqueous solution of sodium and potassium hydroxides in molar ratio 65:35 (to which solution has been added 1 part of common salt to each 100 parts of hydroxides), at a temperature maintained at about 160° F., is delivered through pipe 89 to neutralizer 91 at a rate equivalent chemically to the fatty acid delivery rate through pipe 84.

Neutralizer 91 is constructed along the same general lines as neutralizer 16 shown in Figure 1 and described in Example 1. A continuous stream of soap previously formed by the process is delivered into the mixer through pipes 106 and through a continuous soap cooling device, shown as element 120 in Figure 3, this stream of recirculated soap of reduced temperature having a volume rate about eight times the combined volume rates of the caustic and fatty acids passing through pipes 84 and 89.

Sufficient heat is extracted from the recirculated stream of soap in cooler 120 to reduce the temperature of the soap leaving the neutralizer through line 99, to 140° F. The construction of the soap cooler 120 may be along the same general lines as that of the cooler and neutralizer 51, shown in Figure 2. (The mixer 100 may be replaced, if desired, with a large enclosed heavy-duty mixer having agitator arms attached to the shaft and having staggered holdback members attached to the inner wall, and designed to be completely filled with soap during operation. With soaps of quite low moisture content and heavy consistency, such a design offers advantages over the open type of mixer illustrated as element 100 in Figure 3.)

Soap leaving the process through pipe 117 in this example may be formed into bars, granules, flakes or other desired soap product.

These examples are but a few typical illustrations of practical applications of my process. It is quite obvious that considerable flexibility of conditions and reactants is possible.

As previously stated, the process herein described and claimed is particularly effective in continuously saponifying fatty acids derived from those fats which are commonly employed in the manufacture of detergent soaps for household and industrial use. Such fats, as is well known, include tallow, hog grease or lard, waste greases, palm oil, olive oil and olive oil "foots," coconut oil and palm kernel oil and other similar tropical nut oils rich in glycerides of lauric and myristic acids (the oils of this tropical nut oil class generally being less than 50 per cent of the total mixture of soap-making fats, except in the manufacture of specialty soaps such as bar soaps for use in hard water, soaps intended to give a great profusion of lather, etc.), hydrogenated fish oils and marine animal oils, hydrogenated cottonseed and soybean and other similar temperate zone seed oils, "foots" or "soapstock" from the caustic refining of crude vegetable oils, and others of lesser present day commercial importance. Rosin and synthetic fatty acids derived from petroleum and other organic sources may also be saponified by the present process, and these are included within the scope of the appended claims. Of the alkalies which may advantageously be employed in continuously neutralizing soapmaking fatty acids according to the present process, sodium hydroxide is first in importance, mixtures of sodium hydroxide and potassium hydroxide may be considered next in importance, followed in order by potassium hydroxide, ammonium hydroxide, the alkylamine and alkanolamine hydroxides, and mixtures of these alkaline materials. Carbonates of alkalies yielding water-soluble soaps, such as sodium carbonate, may be used if suitable provision is made for the elimination of at least the greater portion of the carbon dioxide gas which results from the reaction of such carbonates with soapmaking acids.

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

1. In a continuous process of neutralizing soapmaking fatty acids to produce directly, without a drying step, soap of predetermined moisture content between about 12 per cent and 27 per cent, the steps of: intimately mixing proportioned streams of fatty acids and of an aqueous solution of an alkali in the presence of a recirculated and precooled stream of soap previously formed in the process, said aqueous solution having a concentration such that the soap resulting from the neutralization has said predetermined moisture content between about 12 per cent and about 27 per cent; and regulating the temperature of the reactants in the zone of neutralization so that the soap as formed, together with the recirculated soap, is in a pumpable condition, between 130° F. and 215° F., and is above that temperature at which it loses its pasty cohesiveness and becomes tough and resistant to flow as it approaches complete solidification, and is below its atmospheric boiling point.

2. The process of claim 1, in which the proportioned stream of fatty acids is merged with the recirculated stream of previously formed soap ahead of the point at which the proportioned stream of the alkali solution is admixed with the recirculated stream of soap.

3. In a continuous process of neutralizing soapmaking fatty acids without the local overheating of portions of the reacting ingredients, to produce directly, without a drying step, soap of predetermined moisture content between about 12 per cent and 27 per cent, the steps of: intimately mixing, within an inch at most of the cooling surface of an efficient continuous cooling device in which the cooling surface is freed of chilled soap and the contents are agitated at a frequency at least as great as 800 times per minute, proportioned streams of fatty acids and of an aqueous solution of an alkali, said solution having a concentration such that the soap resulting from the neutralization has said predetermined moisture content between about 12 per cent and about 27 per cent, and causing said fatty acids and alkali solution to react in the presence of said cooling surface as aforesaid; and regulating the temperature of the reactants so that the soap as formed is in a pumpable condition, between 130° F. and 215° F., and is above that temperature at which it loses its pasty cohesiveness and becomes tough and resistant to flow as it approaches complete solidi-

fication, and is below its atmospheric boiling point.

4. The process of claim 3, in which a recirculated stream of soap previously formed in the process is continuously passed through said cooling device.

5. In a continuous process of neutralizing soap-making fatty acids to produce directly, without a drying step, soap of predetermined moisture content between about 12 per cent and 27 per cent, the steps of: intimately mixing proportioned streams of a slurry of crystallized fatty acids in molten fatty acids and of an aqueous solution of an alkali, said solution having a concentration such that the soap resulting from the neutralization has said predetermined moisture content between about 12 per cent and about 27 per cent; and regulating the temperature of said fatty acid slurry, in relation to the temperature of said alkali solution, so that the soap as formed is in a pumpable condition, between 130° F. and 215° F., and is above that temperature at which it loses its pasty cohesiveness and becomes tough and resistant to flow as it approaches complete solidification, and is below its atmospheric boiling point.

6. The process of claim 5, in which a recirculated stream of soap previously formed in the process is continuously passed through the fatty acid and alkali mixing zone.

7. In a continuous process of neutralizing soap-making fatty acids without the local overheating of portions of the reacting ingredients, to produce directly, without a drying step, soap of predetermined moisture content between about 18 per cent and about 22 per cent, the steps of: intimately mixing, within an inch at most of the cooling walls of a continuous cooling device in which the cooling surface is freed of chilled soap and the contents are agitated at a frequency at least as great as 800 times per minute, chemically equivalent streams of soap-making fatty acids and of an aqueous solution of caustic soda, said solution having a concentration such that the soap resulting from the neutralization has said predetermined moisture content between about 18 per cent and about 22 per cent, and causing said fatty acids and alkali solution to react in the presence of said cooling surface as aforesaid; and regulating the temperature of the reactants so that the temperature of the soap as formed is between about 130° F. and about 150° F.

8. In a continuous process of neutralizing soap-making fatty acids to produce directly, without a drying step, soap of predetermined moisture content between about 18 per cent and 22 per cent, the steps of: intimately mixing proportioned streams of a slurry of crystallized soap-making fatty acids in molten soapmaking fatty acids and of an aqueous solution of caustic soda, said solution having a concentration such that the soap resulting from the neutralization has said predetermined moisture content between about 18 per cent and about 22 per cent; and regulating the temperature of the reactants so that the temperature of the soap as formed is between about 130° F. and about 150° F.

9. In a continuous process of neutralizing soap-making fatty acids to produce directly, without a drying step, soap of predetermined moisture content between about 18 per cent and about 22 per cent, the steps of: intimately mixing proportioned streams of fatty acids and of an aqueous solution of caustic soda in the presence of a recirculated and precooled stream of soap previously formed in the process, said aqueous solution having a concentration such that the soap resulting from the neutralization has said predetermined moisture content between about 18 per cent and about 22 per cent; and regulating the temperature of the reactants in the zone of neutralization so that the soap as formed, together with the recirculated soap, is in pumpable condition and is between about 190° F. and 215° F.

10. In a continuous process of neutralizing soap-making fatty acids without the local overheating of portions of the reacting ingredients, to produce directly, without a drying step, soap of predetermined moisture content between about 12 per cent and about 27 per cent, the steps of intimately and substantially instantaneously mixing proportioned streams of fatty acids and of an aqueous solution of an alkali while simultaneously withdrawing from these reacting materials at their point of mixing, by heat absorbing means other than the immediately reacting substances in intimate contact with all parts thereof throughout the zone of the reaction, at least the greater part of the heat of reaction, thereby preventing an elevation of the temperature of any portion of the reacting mass sufficient to injure the quality of the resulting soap, said solution of alkali having a concentration such that the soap resulting from the neutralization has said predetermined moisture content between about 12 per cent and about 27 per cent; and regulating the temperature of the reactants so that the resulting soap upon the withdrawal of said heat of reaction is in a pumpable condition, between 130° F. and 215° F., and is above that temperature at which it loses its pasty cohesiveness and becomes tough and resistant to flow as it approaches complete solidification, and is below its atmospheric boiling point.

11. In a continuous process of neutralizing soap-making fatty acids without the local overheating of portions of the reacting ingredients, to product directly, without a drying step, soap predominantly in the beta phase and having a predetermined moisture content between about 12 per cent and about 27 per cent, the steps of: intimately and substantially instantaneously mixing proportioned streams of fatty acids comprising at least 15 per cent of saturated fatty acids having from 16 to 22 carbon atoms per molecule and of an aqueous solution of an alkali consisting predominantly of an alkaline sodium compound while simultaneously withdrawing from these reacting materials at their point of mixing, by heat absorbing means other than the immediately reacting substances in intimate contact with all parts thereof throughout the zone of the reaction, at least the greater part of the heat of reaction, thereby preventing an elevation of the temperature of any portion of the mass sufficient to injure the quality of the resulting soap, said solution of alkali having a concentration such that the soap resulting from the neutralization has said predetermined moisture content between about 12 per cent and about 27 per cent; and regulating the temperature of the reactants so that the resulting soap upon the withdrawal of said heat of reaction is in a pumpable condition, between 130° F. and 215° F., and is above the temperature at which it loses its pasty cohesiveness and becomes tough and resistant to flow as it approaches complete solidification, and is within the temperature range in

which the beta phase is formed in substantial amount upon agitating.

12. In a continuous process of neutralizing soap-making fatty acids without the local overheating of portions of the reacting ingredients, to produce directly, without a drying step, soap predominantly in the omega phase and having a predetermined moisture content between about 12 per cent and about 27 per cent, the steps of: intimately and substantially instantaneously mixing proportioned streams of fatty acids and of an aqueous solution of an alkali while simultaneously withdrawing from these reacting materials at their point of mixing, by heat absorbing means other than the immediately reacting substances in intimate contact with all parts thereof throughout the zone of the reaction, at least the greater part of the heat of reaction, thereby preventing an elevation of the temperature of any portion of the mass sufficient to injure the quality of the resulting soap, said solution of alkali having a concentration such that the soap resulting from the neutralization has said predetermined moisture content between about 12 per cent and about 27 per cent; regulating the temperature of the reactants so that the resulting soap upon the withdrawal of said heat of reaction is in a pumpable condition, between 130° F. and 215° F., above its complete melting point and below its atmospheric boiling point; and cooling the resulting soap from fully molten to fully solidified condition without agitation.

13. The continuous process of neutralizing soap-making fatty acids without the local overheating of portions of the reacting ingredients, to produce directly, without a drying step, a continuous stream of soap of uniform composition and of predetermined moisture content between about 12 per cent and 27 per cent, which comprises: intimately and substantially instantaneously mixing proportioned streams of fatty acids and of an aqueous solution of an alkali while simultaneously withdrawing from the reacting materials at their point of mixing, by heat absorbing means other than the immediately reacting substances in intimate contact with all parts thereof throughout the zone of the reaction, at least the greater part of the heat of reaction, thereby preventing an elevation of the temperature of any portion of the reacting mass sufficient to injure the quality of the resulting soap, said solution of alkali having a concentration such that the soap resulting from the neutralization has said predetermined moisture content between about 12 per cent and about 27 per cent; regulating the temperature of the reactants so that the resulting soap upon the withdrawal of said heat of reaction is in a pumpable condition, between 130° F. and 215° F., and is above that temperature at which it loses its pasty cohesiveness and becomes tough and resistant to flow as it approaches complete solidification, and is below its atmospheric boiling point; completing the neutralization reaction and the blending of the resulting soap in a separate chamber, having a soap capacity at least equivalent to the amount of soap produced in said process each 30 minutes, by continuously discharging the soap resulting from the preceding two steps into said separate chamber and therein continuously mixing and recirculating said soap; and continuously withdrawing soap from said final neutralization and blending step at a rate substantially equivalent to the rate of production of soap in said process.

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