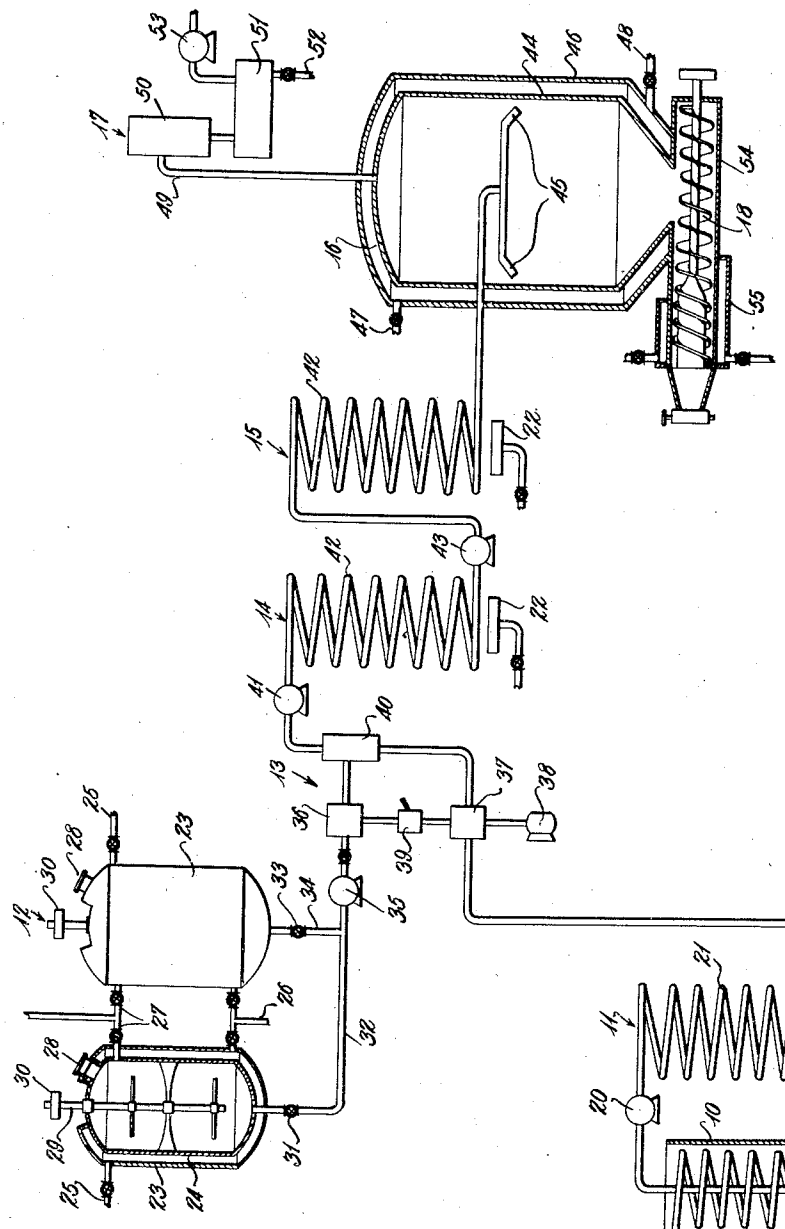
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PROCESS OF MAKING SOAP

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Inventor

Benjamin H. Thurman

By Harris, Kiech, Foster & Harris

Attorneys

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PROCESS OF MAKING SOAP

Benjamin H. Thurman, Bronxville, N. Y., assignor to Refining, Inc., Reno, Nev., a corporation of Nevada

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3 Claims. (Cl. 260—416)

This invention relates to a process of making soap, and more particularly to a process employing concentrated solutions of alkali as the saponifying material.

Heretofore, in soap making processes, the alkalies, particularly caustic alkalies such as caustic soda, have been employed in relatively dilute concentration, as compared to those contemplated by the present invention, for saponifying materials such as glycerides, fatty acids, abedic acids, and higher fatty acid esters of higher fatty alcohols such as, for example, sperm oil. Such materials have ordinarily been saponified by the conventional batch process in large open soap making kettles. The temperature which could be employed was not greatly in excess of the boiling point of water. Thus relatively dilute solutions of akalies were necessarily employed in order to provide sufficient water to maintain the soap in fluid condition. Even with soap making processes employing closed kettles, caustic soda solutions above 50% caustic soda content have not been employed, as this amount of caustic soda is approximately all that can be dissolved in water at ordinary temperatures. The present invention is directed to a process of making soap in which caustic soda solutions substantially above 50% concentration and ranging up to concentrations of 35% to 95% are employed. A large portion of known soap making processes is concerned with removing water from the soap produced, and all of such processes involve heating large quantities of water present during the saponification reaction, thus wasting large amounts of heat. According to the present invention, concentrated caustic alkali solutions (materially above 50% concentration) are employed, thus reducing the amount of material which must be heated, and also reducing the amount of water which must be separated from the soap. The present invention finds its chief utility in a process in which glycerine or other volatile materials such as higher fatty alcohols are continuously removed from the soap so as to produce substantially anhydrous soap while recovering the volatile materials admixed with lesser amounts of water. In such processes, any water in the process must also be evaporated, and the present invention thus markedly reduces the amount of heat necessary for such evaporation and the amount of cooling necessary to condense water.

It is, therefore, an object of the present invention to provide a process of making soap in which caustic alkali solutions in concentrations substantially above 50% are employed.

Another object of the invention is to provide a process of producing soap and recovering volatile materials liberated in the process in which the amount of water evaporated and condensed is markedly reduced.

A further object of the invention is to provide a continuous process of producing soap in which concentrated solutions of saponifying alkali are continuously mixed with the saponifiable material under high temperature conditions.

A still further object of the invention is to provide a process of saponifying glycerides of fatty acids and directly recovering relatively concentrated glycerine.

Other objects of the invention will appear in the following description of the preferred embodiment thereof. This description is given in connection with the attached drawing, which diagrammatically shows an apparatus capable of carrying out the present invention.

Referring more particularly to the drawing, 10 indicates a source of supply, such as a tank, for the saponifiable material; 11 indicates a heating device for preheating the saponifiable material before admixture with the saponifying materials; 12 indicates a device for forming concentrated solutions of alkali; 13 indicates in general a proportioning and mixing system for continuously mixing the preheated saponifiable material and alkali; 14 and 15 indicate heating devices for the resulting mixture; 16 indicates a vapor separating chamber for separating volatile materials from the soap; 17 indicates in general a condenser system for condensing such volatile materials and maintaining a vacuum in the vapor separating chamber 16; and 18 indicates a conveyor for removing soap from the vapor separating chamber without breaking the vacuum and cooling the soap.

The tank 10 for the saponifiable material may include a heating coil or other heating device 19 for rendering the saponifiable material flowable, if necessary. It is preferred not to heat such material in large amounts and in contact with the atmosphere to temperatures much above that necessary for enabling the same to be pumped. A pump 20 may be provided for withdrawing saponifiable material from the tank 10 and forcing the same through the heating device 11, which may include a heating coil 21 and a burner 22 for liquid or gaseous fuel.

The device for forming concentrated solutions of alkali may include one or more receptacles

23 provided with heating jackets 24, or other heating means, in order to heat materials contained therein. Provision is made for circulating any desired heating medium, such as steam or heated mineral oil, through the jackets 24 by means of pipes 25 and 26. The receptacles 23 are preferably closed and capable of sustaining substantial internal pressure, but in certain modifications of the present process, open vessels may be employed. Water may be introduced into the receptacles 23 through pipes 27, and, likewise, alkali may be introduced in measured amounts through removable closure members 28. Each receptacle 23 is also provided with an agitator 29, which may be driven from any suitable source of power through a pulley 30. By mixing the alkali, such as caustic soda, with water under high temperature conditions, and preferably under pressure to prevent evaporation of the water, extremely strong fluid solutions of alkali may be produced. For example, caustic soda solutions ranging from substantially above 50% caustic soda to solutions containing only a small percentage of water may be prepared.

For a continuous process, the receptacles 23 may be employed alternatively to deliver material to the process from one of the receptacles while a solution is being prepared in the other receptacle. Thus, by opening the valve 31 in the pipe 32 and closing the valve 33 in the pipe 34, a solution of the desired concentration may be pumped from one of the receptacles by a pump 35 and delivered to a proportioning pump 36 forming part of the proportioning and mixing system 13. Likewise, the heated saponifiable material from the heating coil 31 may be delivered to the proportioning pump 37. The proportioning pumps 36 and 37 may be driven by a variable speed motor 38 and a variable speed device 39 may be interposed between said pumps, so that desired amounts and proportions of saponifiable material and alkali may be delivered to the mixer 40. The particular proportioning apparatus shown is merely by way of example, and any other type of proportioning apparatus capable of delivering accurately proportioned streams to the mixer 40 may be substituted therefor. Also, in practicing the process, the heating device 11 may be positioned between the proportioning pump 37 and the mixer 40 instead of the position shown. Also a similar heating device for the alkali solution may be employed either before or after the proportioning pump 36, if it is desired to prepare the alkali solution at a temperature below that at which it is delivered to the mixer 40.

The mixer 40 is preferably a flow mixer of any suitable type which will thoroughly admix the stream of saponifiable material and alkali. It is usually sufficient to merely inject a stream of one material at right angles into a floating stream of the other, but the mixer disclosed in the copending application of Benjamin H. Thurman, Serial No. 190,673 filed February 15, 1938, and entitled Proportioning apparatus, has been found to be extremely efficient, and is preferred. The resultant mixture may be forced by a pump 41 through the heating device 14, which may include a heating coil 42 and a burner 22. Preferably one or more additional heating devices 15 also including a coil 42 and burner 22 are provided with an associated pump 43 for forcing the mixture through the heating coil 42.

The heating devices 14 and 15 are employed to give sufficient time for substantially complete reaction and also to increase the temperature of the mixture during flow, if necessary, to cause said substantially complete reaction and maintain the resultant soap in liquid form. Preferably, this temperature is sufficiently high to cause the soap to be in molten form when anhydrous, after water and other vaporizable materials have been removed from the soap in the vapor separating chamber 16. The pressure and temperature in at least the latter heating device 15 are also desirably adjusted to cause all, or a substantial part, of the vaporizable materials to be in vapor form when delivered into the vapor separating chamber 16.

The mixture from the heating device 15 is preferably discharged against the wall 44 of the vapor separating chamber by nozzles 45 positioned adjacent the wall 44. The wall 44 is maintained at a temperature above the melting point of the substantially anhydrous soap produced by means of a heating jacket 46 surrounding the vapor separating chamber 16, and through which any desired heating medium, such as heated mineral oil, may be circulated by pipes 47 and 48. By thus maintaining the temperature of the wall 44 above the melting point of the anhydrous soap, the soap will flow down the wall 44 in thin films, thus effecting the liberating of vapors entrained in the soap and formed in the vapor separating chamber 16. A substantially unimpeded path for the vapors is thus provided inwardly and upwardly through the vapor separating chamber to the vapor withdrawal pipe 49 through which vapors are delivered to the condenser 50 forming part of the condensing system 17. The condensed vaporizable material is collected in a receiver 51, from which it may be withdrawn through a pipe 52 by any suitable means such as a pump (not shown). A vacuum pump 53 connected to the receiver 51 is provided for maintaining a vacuum in the vapor separating chamber 16 and condensing system 17.

Molten anhydrous soap flows down the wall 44 of the vapor separating chamber 16 into the casing 54 of the conveyor 18 and is continuously removed from the vapor separating chamber. The conveyor 18 is preferably provided with a cooling jacket 55 for cooling the soap withdrawn from the evaporating chamber. A series of one or more additional conveyors (not shown), connected to the conveyor 18, may be employed for further cooling, as disclosed in the copending application of Benjamin Clayton and Benjamin H. Thurman, Serial No. 119,168, filed January 5, 1937. The vapor separating chamber and conveyor system employed in the present invention may be the same as the apparatus disclosed in said application.

As before stated, a 50% sodium hydroxide solution is substantially the greatest concentration that can be prepared at ordinary temperatures; that is, approximately 100 parts of caustic soda can be dissolved in 100 parts of water at 68° F., whereas 347 parts of caustic soda can be dissolved in a like amount of water at 212° F. to produce a solution of approximately 78% concentration. By mixing under pressure, extremely large amounts of caustic soda can be thus dissolved, and solutions containing only, for example, 5 to 10% water can be prepared. By mixing a stream of such concentrated caustic soda heated to a temperature of, for example, 200° F. to 500° F., depending upon the saponifiable material employed, with a stream of glycerides of fatty acids,

fatty acid esters, or other saponifiable material preheated to temperatures, for example, between 400° F. and 620° F., extremely rapid saponification can be accomplished, and a soap solution produced which is liquid at the temperature of the mixture. Additional heat is usually necessary to vaporize the glycerine and water if glycerides are employed as the saponifiable material, in which case as many heating devices 14 or 15 may be employed as are necessary to impart such heat. The same is true when employing higher fatty acid esters of higher fatty acid alcohols, such as sperm oil, in which case vaporization of higher fatty alcohols liberated requires additional heat. Also, some time of reaction is usually required with such materials, and heating devices such as 14 or 15 provide time for the completion of the reaction.

When saponifying fatty acids, the reaction is almost instantaneous, and the temperature of mixing must be sufficient to form a fluid soap mixture. Thus the alkali solution must usually be preheated to at least 400° F. and the saponifiable material preheated to the temperature above given, such that the temperature of mixing is between approximately 400° F. and 600° F. As water is substantially the only thing to be vaorized, the material can be delivered directly to the vapor separating chamber, and the heating devices 14 and 15 omitted. Commercial bar or powdered soaps contain from 10% to 30% water, and, by adjusting the water content of the concentrated alkali solution when saponifying substantially pure fatty acids, the resulting mixture may be delivered directly to the conveyor 18, or other cooling device, to directly produce a soap having the requisite amount of water for such bar or powdered soap. Thus, a bar of soap may be extruded directly from the conveyor system and cut in to bars or otherwise treated to produce soap in condition to be directly marketed as a detergent.

If it is desired to render the soap anhydrous, or recover glycerine or higher fatty alcohols liberated in the process, the mixture of soap and volatile materials should be delivered to the vapor separating chamber at a temperature between approximating 450° F. and 620° F., depending upon the type of material being saponified. That is, the mixture should be sufficiently hot that the soap is produced in molten anhydrous form after the volatiles have been removed. It is also desirable to maintain a sufficiently low pressure in at least the last heating device 15 to cause the vaporizable material to be partly, or all, in vapor form when discharged into the vapor separating chamber. Thus, pressures as low as 50 to 100 pounds per square inch can be employed in the latter part of the heating device. Since the temperature throughout the portion of the system in which saponifiable materials and caustic are in contact or soap is present, is sufficiently high to render the materials liquid, relatively low pressures, for example 150 to 300 pounds per square inch, can be utilized, even in the entrance portions of the heating devices. If viscous mixtures are encountered, the addition of small amounts of inert liquids, such as kerosene, can be added to the saponifiable material. Such liquids will be vaporized and separated from the soap in the vapor separating chamber. Since the concentrated alkali solutions contain less water than those heretofore used, less heat is utilized in the system to maintain the requisite temperatures and vaporize water from the soap.

By increasing the length of the heating devices 14 or 15 and thus the length of time the materials are subjected to high temperatures, the Varrentrapp reaction may be obtained to any desired extent. This reaction involves the combination of caustic alkali with unsaturated fatty acid radicals to liberate hydrogen and produce fatty acid radicals of less unsaturation and having a smaller number of carbon atoms than the original unsaturated fatty acid radicals. Thus a more stable and harder soap can be produced. The presence of water in the system, however, retards this reaction as compared to that which would be obtained in an anhydrous system. The materials in the process are maintained out of contact with the air when at high temperatures, and are maintained in sufficient motion to prevent local overheating with resultant pyrolytic decomposition, such that the soap or other fatty materials are not damaged.

While the preferred embodiments of this invention are disclosed herein, it is understood that the details thereof may be varied within the scope of the following claims.

I claim as my invention:

1. The process of making soap and recovering concentrated glycerine directly therefrom, which comprises, mixing a stream of saponifiable material consisting essentially of glycerides of fatty acids with a stream of alkali solution containing substantially in excess of 50% of alkali, preheating said stream of saponifiable material to a temperature between approximately 400° F. and 620° F. before admixture with said alkali solution, preheating said alkali solution to a temperature between approximately 200° F. and 500° F. before admixing the same with said saponifiable material, adding sufficient additional heat to a flowing stream of said mixture to substantially completely react said alkali and said saponifiable material to form soap and liberate glycerine, thereafter discharging the resulting mixture containing soap and glycerine into a vapor separating zone while at a temperature above the melting point of said soap when anhydrous, withdrawing and condensing glycerine vapors from said zone at a rate sufficient to maintain a vacuum in said zone, and continuously removing soap from said zone without breaking said vacuum.

2. The process of making soap, which comprises, mixing a stream of saponifiable material with a stream of alkali solution containing substantially in excess of 50% of alkali, preheating said stream of saponifiable material to a temperature between approximately 400° and 620° F. before admixture with said alkali solution, preheating said alkali solution to a temperature between approximately 200° and 500° F. before admixing the same with said saponifiable material, adding sufficient additional heat to a flowing stream of the resulting mixture to substantially completely react said saponifiable material and alkali to form soap, thereafter discharging the resulting mixture containing soap and vaporizable material into a vapor separating zone while at a temperature above the melting point of said soap when anhydrous, withdrawing and condensing vapors of vaporizable material from said zone at a rate sufficient to maintain a vacuum in said zone and continuously removing soap from said zone without breaking said vacuum.

3. The process of making soap, which comprises, mixing a stream of saponifiable material consisting essentially of fatty acids with a stream

of alkali solution containing substantially in excess of 50% alkali, preheating said stream of saponifiable material to a temperature between 400° and 620° F. before admixture with said alkali solution, preheating said alkali solution to a temperature between approximately 200° and 500° F. before admixing the same with said saponifiable material, said saponifiable material and alkali being preheated to a temperature sufficiently high to produce a flowable mixture, substantially completely reacting said alkali and said saponifiable material while in a flowing stream to form soap, thereafter discharging the resulting mixture into a vapor separating zone while at a temperature above the melting point of said soap when anhydrous, withdrawing and condensing vapors from said zone at a rate sufficient to maintain a vacuum in said zone, and continuously removing soap from said zone without breaking said vacuum.

BENJAMIN H. THURMAN.