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MANUFACTURE OF SOAP AND GLYCERIN

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This invention relates to improvements in the process of making soap, and particularly to methods for separating glycerin from soap, and for cooling and hydrating the resulting soap.

One object of my invention is to provide a continuous and economical procedure for separating glycerin from soap in the manufacture of same.

Another object is to provide a continuous procedure for cooling and hydrating highly heated anhydrous soap.

Another object is to remove odors from soap.

In the usual process for manufacture of soap and glycerin, the fat is saponified by boiling with a rather dilute aqueous solution of caustic soda or other saponifying agent, which in the first step results in a mixture of soap and glycerin with considerable water. In the second step, salt is added causing a separation into two layers, the upper layer consisting of soap containing about thirty per cent moisture, and the lower layer consisting of an aqueous solution of glycerin and salt with any excess of saponifying agent. The soap layer is then washed several times to remove more glycerin, and finally cooled to the desired temperature for further processing, usually by natural radiation, or sometimes by artificial cooling. The glycerin solution, usually containing only about five to fifteen per cent of glycerin is purified and evaporated to obtain the crude glycerin which is then further purified and concentrated by distillation. Several days are usually required to make a batch of soap in this way on a commercial scale. Such soap contains much of the odor-ingredients characteristic of the fat used, which may be very objectionable in the product if low grade fats were used.

Attempts have been made to shorten this procedure. It is known, for example, that a rapid reaction between saponifiable fatty matter and molten caustic alkalis or concentrated solutions of same can be had with the production of soap and glycerin, provided quite high temperatures are maintained; it has been suggested that the glycerin be distilled off during such high temperature saponification by the use of highly superheated steam, leaving anhydrous, glycerin-free, soap in a molten condition at a high temperature. It has also been suggested that a completely saponified product consisting of soap, glycerin, and water, be sprayed under certain high temperature conditions without the aid of superheated steam to allow the water and most, or all, of the glycerin to volatilize and obtain as a result anhydrous soap in form of a powder having a high temperature.

A completely anhydrous soap is rarely desired for commercial use, so that when glycerin-free anhydrous soap at high temperatures, either in molten condition or in powder form, has been produced or described in the past, various methods of hydrating same have been proposed, but all such proposals, as far as I am aware, are limited in respect to moisture range obtainable and temperature of product; these limits are not always what is required for further processing or commercial use, and serious operating difficulties are also involved.

So far as I have been able to determine, no one has suggested a practical, complete and continuous procedure for converting saponifiable fat into glycerin-free soap, and concentrated glycerin, with the soap free from odor even when made from a low grade fat, and delivered continuously at any desired moisture content and at substantially any desired temperature. Then, further, no one has provided a continuous method, so far as I have determined, for adding a controlled amount of water to a flowing stream of molten anhydrous soap to convert it into hydrated soap containing any desired amount of moisture for further processing or for commercial use, and at the proper temperature for such further processing.

My process consists briefly in saponifying fat, (this word meaning any saponifiable fat or fatty oil), with a concentrated solution of alkali hydroxide at a high temperature and superatmospheric pressure, introducing the highly heated saponified mass into a suitable distilling chamber with a countercurrent of superheated steam under vacuum, whereby the glycerin, water, and odors are distilled off, leaving the soap in a glycerin-free anhydrous condition at a temperature high enough to keep same molten, and capable of being pumped, then subjecting the molten anhydrous soap to a superatmospheric pressure, mixing a flowing current of same with a flowing current of water of regulated temperature and amount while under pressure, and finally discharging the soap and water mixture into an atmosphere of lower pressure, which may be atmospheric pressure, or higher, or lower. By proper choice of the temperature and amount of water used, and the pressure of the atmosphere into which the soap and water mixture is discharged, a product can be obtained having any desired moisture content up to that of the "middle soap" phase, or about thirty-five per cent, and at any desired temperature corresponding to the boiling point of water at the pressure used.

In carrying out my process, I first saponify the fatty matter such as tallow or other saponifiable fat or oil, or a mixture of same, with a saponifying agent such as sodium hydroxide solution, at a high temperature and pressure, keeping the water in the mixture at a low point, preferably not more than ten to twelve per cent

for reasons explained below. With such low percentages of moisture a high temperature such as 180°–200° C. or higher is necessary to keep the saponified mixture in fluid and pumping condition. I heat the saponified mass containing soap, glycerin, and water, further if necessary, to bring the temperature up to about 600° F. (316° C.) in case of a soda soap, under sufficient pressure to prevent volatilization of the water or glycerin in the mixture, a pressure of about 1550 pounds per square inch being required at a temperature of 600° F. It is preferable, however, to obtain the desired final temperature by heating sufficiently the ingredients before saponification, as the heat transfer is more readily effected then, and there is less danger of scorching. The minimum temperature required at this point cannot be stated with exactness for all cases as it will vary with soaps made from different fatty materials and with varying content of glycerin and water, but in general it should be sufficiently high to store in the mass most, but not necessarily all, of the heat necessary for subsequent volatilization of the glycerin and water without permitting the temperature of the resulting anhydrous soap to be thereby reduced below its flowing point, (about 446° F. or 230° C. for most common soaps), but on the other hand the original temperature should not be high enough to injure the soap or the glycerin. 600° F. is a satisfactory temperature for most soaps containing not more than ten to twelve per cent of moisture. Lower temperatures are sufficient with potash soaps than with soda soaps because of the lower melting points of such soaps at this stage. The presence of excess water will make it necessary to supply correspondingly more heat to volatilize same; hence, I keep the percentage of water in the mass at a low point, preferably about ten to twelve per cent, which is the result of using a caustic soda solution of about fifty per cent concentration for the saponification. The high pressure of about 1550 pounds is only needed in the heating system and the pipe leading to the glycerin distilling chamber, so the total volume of material under this pressure can be kept very small as a safety precaution. Heating may be accomplished by passing the mass through a coil of pipe suitably heated by any known means.

To separate the glycerin from the soap, this heated mixture is discharged into the top of a suitable chamber or still maintained under a vacuum, or low absolute pressure, with a countercurrent of highly superheated steam, so as to cause substantially all the glycerin, water, and odors in the hot saponified mass to volatilize while the soap itself passes downward to the bottom in the form of molten anhydrous soap. For best results in distillation of glycerin and removal of odors the absolute pressure in this chamber should not be more than about 25 mm. of mercury, preferably not more than 5 mm. of mercury. This chamber is preferably a tower, containing a series of baffles, trays, or plates, over which the soap flows in thin layers to assist in the complete separation of the glycerin, water, and odors from the soap during its passage from the top to the bottom of the chamber. It is also possible to obtain fairly good results by spraying the saponified mass into the top of a chamber without baffles or plates and allowing the soap to fall downward through the ascending superheated steam in form of small drops.

With a high initial temperature of the saponified mass, by avoiding an unnecessary excess of water in same, and by the use of a sufficiently low absolute pressure in the distilling chamber, substantially all of the glycerin, water, and odors, can be caused to volatilize with the use of a moderate and entirely practical amount of superheated steam and without allowing the temperature of the anhydrous soap to be thereby reduced below its flowing point, although the volatilization naturally absorbs heat and causes a considerable reduction in temperature.

When the soap mass containing glycerin and water at a high temperature and pressure, is suddenly subjected to a low absolute pressure as described, the particles constituting the mass burst or shatter due to the sudden volatilization of the glycerin and water within the particles. This action is entirely different in its mechanism and results from spraying a similar mass at a temperature below the volatilization point into a surrounding atmosphere of highly superheated steam or other gas in the attempt to raise the temperature after spraying to the volatilization point of the glycerin and the water. In the latter case it is impracticable to transfer heat from the surrounding atmosphere fast enough, at the low absolute pressure required to volatilize all the glycerin, while keeping the soap at a fluid temperature, during the passage from top to bottom of the chamber.

It is my purpose therefore to store up within the mass most of the requisite heat in order to produce a disintegrating effect when introduced into the low pressure chamber, thus greatly facilitating the complete volatilization of glycerin, water, and odors. The function of the superheated steam is to supply any additional heat needed to complete the volatilization of glycerin, water, and odors, and to prevent the temperature of the glycerin-free anhydrous soap from dropping below its temperature of fluidity. It incidentally assists also by reducing the partial vapor pressure of the glycerin.

The amount of superheated steam required will vary according to conditions. Assuming, for example, a soap made by saponifying tallow with caustic soda solution of about fifty per cent concentration, heated to 600° F. before introducing into the distilling chamber having an absolute pressure of one inch of mercury, I find that about 7 pounds of steam superheated to 750° F. at a pressure of 5 pounds per square inch are needed per 100 pounds of the saponified mass. Under these conditions the heat previously stored up within the mass plus the heat derived from the superheated steam in the distilling chamber will suffice to volatilize substantially all of the glycerin, water, and odors, and keep the resulting anhydrous soap at a fluid temperature.

It is important in this operation to keep all the materials used, including the steam, and also the apparatus itself free from air or other oxidizing agents, and to pass the mass continuously and rapidly through the apparatus so that it is not subjected to the high temperature for more than a very few minutes, in order to avoid darkening of the soap and decomposition of the glycerin.

The glycerin vapors issuing from the apparatus are separated from the steam by fractional condensation in the known way to obtain distilled glycerin in concentrated form.

The hydration and cooling of the molten anhydrous soap thus freed from glycerin, water and odors is accomplished by pumping it continuously from the aforementioned distilling chamber,

raising its pressure to a point sufficient to prevent the volatilization of the water which is to be added, and then conducting a flowing current of the molten soap under this pressure in a pipe, and a flowing current of water in another pipe, to a point where the two pipes unite, so that a thorough mixing of the ingredients occurs. This mixing may take place just before entering a nozzle from which the mixture is sprayed into a chamber in which suitable atmospheric pressure conditions can be maintained, or it may take place within the nozzle itself, or just as the soap and water issue from the nozzle, depending on the type of nozzle used. By proper regulation of the temperature and amount of the water, and of the pressure in the spray chamber, a product of practically any desired moisture content can be obtained at any reasonable temperature. Soaps in the "middle soap" phase, (commonly about 35 to 70% moisture at 212° F.), are however very viscous and difficult to handle, but such moisture contents are outside the usually desired ranges. The temperature of the resulting soap product depends on the pressure in the spraying chamber because the heat in the mass will cause the volatilization of a portion of the water used at the temperature corresponding to the boiling point of water at the pressure in the spraying chamber, provided only that sufficient water be present. Water not thus volatilized, if any, will remain in the soap.

It is thus obvious that by using a properly limited amount of water the heat of the soap may cause all of the water to be volatilized in the spraying chamber, resulting in the production of anhydrous soap at a temperature depending on the amount of water used and the pressure in the spraying chamber, which may be at atmospheric pressure, or above, or below same.

The following table shows examples of the calculated quantity of water required to yield soap products of various moisture contents at various temperatures. These calculations are only approximately correct, as for purposes of simplicity certain factors have been disregarded or are not accurately known. The following assumptions are made in these calculations.

Temperature of molten anhydrous soap=250° C. (482° F.)
Specific heat of anhydrous soap=.70.
Temperature of water used=20° C. (68° F.)
Latent heat of volatilization of water at 212° F.=970 B. t. u.
Latent heat of volatilization of water at 160° F.=1002 B. t. u.

	Anhydrous soap	Water			Spray chamber pressure	Soap product	
		Added	Volume	Remaining		Temperature	Moisture
					Inches	*°F.*	*Percent*
A	100	17	17	0	30	212	0
B	100	20.6	20.6	0	9.6	160	0
C	100	55.5	12.5	43	30	212	30
D	100	60	17	43	9.6	160	30
E	100	36.7	17.6	19.1	9.6	160	15

In the above table, A shows that under the assumed conditions 100 pounds of soap plus 17 pounds of water, when mixed and sprayed into a chamber at atmospheric pressure (30 inches mercury), will yield a soap product containing no moisture and having a temperature of 212° F. All of the water added will have volatilized and the latent heat of volatilization has been the main factor in cooling the soap to 212° F. B shows that to obtain anhydrous soap at 160° F. 20.6 pounds of water are required and the mixture is sprayed into a chamber having an absolute pressure of 9.6 inches of mercury. C shows a soap product containing thirty per cent of moisture, having a temperature of 212° F., (thus corresponding to the usual form of kettle boiled soap), made by the use of 55.5 pounds of water. D shows production of the same grade of soap at a temperature of 160° F., which results from using 60 pounds of water and spraying into an atmosphere with an absolute pressure of 9.6 inches. E shows that a soap product containing fifteen per cent of moisture (approximately the amount desired in the manufacture of milled toilet soap) may be obtained by using 36.7 pounds of water and spraying into a chamber having an absolute pressure at 9.6 inches with a resulting temperature of 160° F. In all the above cases it should be noted that at least a portion of the added water volatilizes due to the heat stored up in the molten anhydrous soap, and the latent heat of volatilization of the water is an important factor in the cooling of the soap. By suitable modifications of the temperatures and proportions of soap and water used, and the absolute pressure in the spraying chamber, practically any desired moisture content and temperature of product can be obtained.

It is thus seen that I have provided a continuous procedure for converting saponifiable fat into a soap product having any desired moisture content from anhydrous soap up to the "middle soap" range, and at practically any temperature required, either for immediate packaging or for further processing into other forms such as bar soap, flakes, powders or milled toilet soap, etc., and the soap is substantially free from odor even when made from fatty materials of low grade which originally had objectionable odors. Built soaps such as are desired for laundry use are obtained by adding suitable builders to the water used for hydrating and cooling. Glycerin is also obtained in concentrated form as one of the products of my process.

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

1. In combination with the step of producing molten anhydrous soap by distillation of water and glycerin from a highly heated soap, the step of cooling the soap from an anhydrous molten condition at a high temperature to an anhydrous condition at a lower temperature, which comprises continuously mixing a flowing current of anhydrous molten soap with a flowing current of liquid water and discharging the soap and water mixture into an atmosphere having a pressure sufficiently below the original pressure of the said mixture to cause volatilization of the water with absorption of heat from said mixture, the amount of added water being adjusted with relation to the total heat of the mixture and the reduced atmospheric pressure into which it is to be discharged so that the heat absorbed from the mixture by volatilization of the water at said reduced pressure will cool the soap and leave substantially no water unvolatilized.

2. A process for cooling soap from an anhydrous molten condition at a high temperature to an anhydrous condition at a lower temperature, which comprises continuously mixing a flowing current of anhydrous molten soap with a flowing current of liquid water and discharging the soap and water mixture into an atmosphere having a

pressure sufficiently below the original pressure of the said mixture to cause volatilization of the water with absorption of heat from said mixture, the amount of added water being adjusted with relation to the total heat of the mixture and the reduced atmospheric pressure into which it is to be discharged so that the heat absorbed from the mixture by volatilization of the water at said reduced pressure will cool the soap and leave substantially no water unvolatilized.

3. In combination with the step of producing molten anhydrous soap by distillation of water and glycerin from a highly heated soap, the step of simultaneously cooling the said molten soap to a desired temperature and hydrating the same to a desired point, which comprises continuously mixing a flowing current of anhydrous molten soap with a flowing current of liquid water under a pressure sufficient to prevent the water from volatilizing, and discharging the soap and water mixture into an atmosphere of lower pressure corresponding substantially to the vapor pressure of water at the desired temperature to which the soap is to be cooled, the amount of water added being sufficient to provide an excess over the amount desired in the final hydrated product and being adjusted with relation to the total heat in the soap and water mixture so that all of the said excess water volatilizes when the mixture is subjected to the said reduced atmospheric pressure with absorption of heat from the mixture due to the latent heat of volatilization of the excess water, thereby cooling the remaining hydrated soap from substantially the temperature of mixing to the desired temperature.

4. A process for simultaneously cooling anhydrous molten soap to a desired temperature and hydrating same to a desired point, which comprises continuously mixing a flowing current of anhydrous molten soap with a flowing current of liquid water under a pressure sufficient to prevent the water from volatilizing and discharging the soap and water mixture into an atmosphere of lower pressure corresponding substantially to the vapor pressure of water at the desired temperature to which the soap is to be cooled, the amount of water added being sufficient to provide an excess over the amount desired in the final hydrated product and being adjusted with relation to the total heat in the soap and water mixture so that all of the said excess water volatilizes when the mixture is subjected to the said reduced atmospheric pressure with absorption of heat from the mixture due to the latent heat of volatilization of the excess water, thereby cooling the remaining hydrated soap from substantially the temperature of mixing to the desired temperature.

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