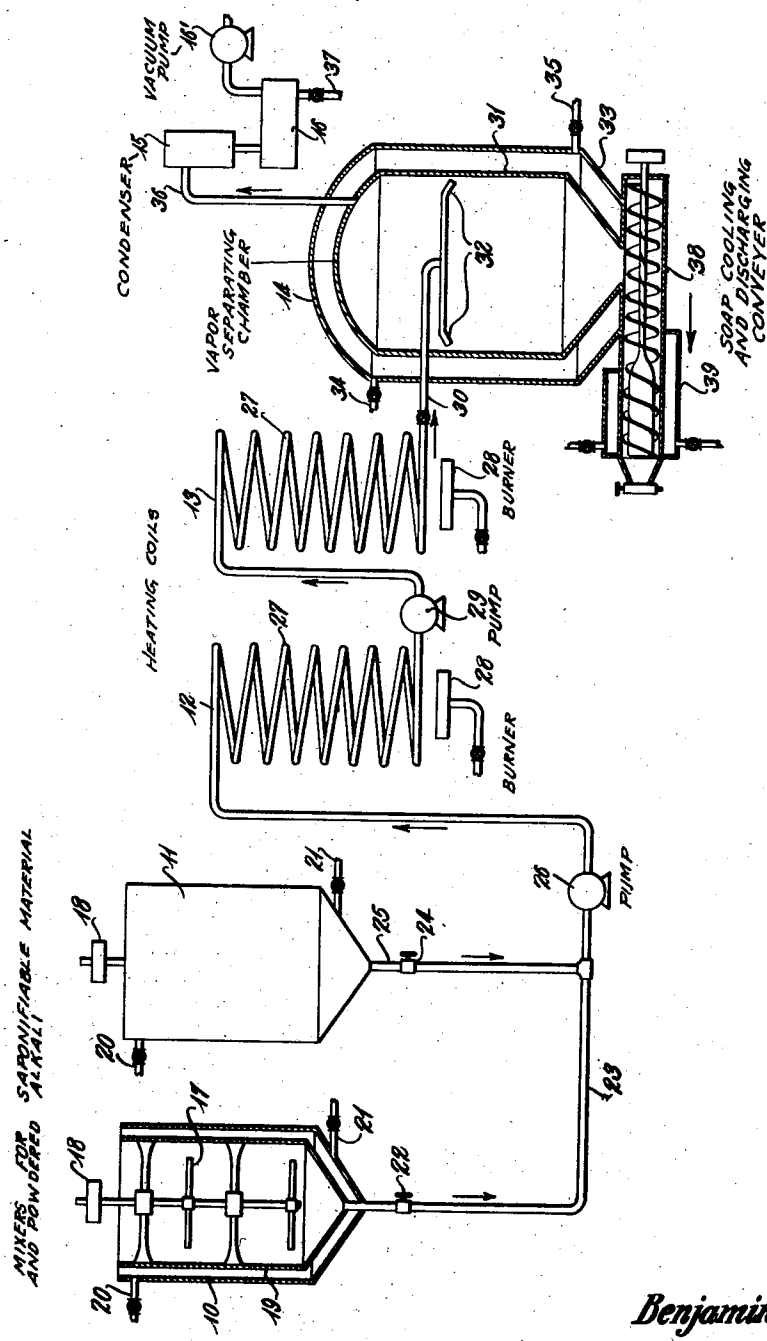
May 13, 1941. B. H. THURMAN 2,242,187

PROCESS OF MAKING SOAP

Filed March 5, 1938



Inventor

Benjamin H. Thurman

By Kerr, Kiech, Foster & Kerr

Attorneys

Patented May 13, 1941 2,242,187

UNITED STATES PATENT OFFICE

2,242,187

PROCESS OF MAKING SOAP

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

Application March 5, 1938, Serial No. 194,222

10 Claims. (Cl. 260—413)

This invention relates to a process of making soap, and more particularly to a process of making anhydrous soap and recovering volatile materials therefrom.

It is an object of the invention to provide a novel process of making anhydrous soap.

Another object of the invention is to provide a process of making soap in which powdered alkali is employed.

Another object of the invention is to provide a process of making soap in which powdered anhydrous alkali is caused to react directly with saponifiable materials containing combined volatiles to form anhydrous soap and liberate said volatiles in recoverable form.

Another object of the invention is to provide a process of making soap in which water is substantially absent from the process and substantially pure and anhydrous glycerine is continuously recovered.

A further object of the invention is to provide a continuous process of causing molten anhydrous caustic alkali to react with glycerol esters of higher fatty acids to form anhydrous soap and liberate anhydrous glycerine.

A still further object of the invention is to provide a continuous process in which fatty materials consisting essentially of glycerides of fatty acids are caused to react with anhydrous caustic soda to produce anhydrous soap separately from anhydrous glycerine.

Other objects and advantages of the invention will appear in the following description of a preferred embodiment of the invention made in connection with the attached drawing, which is a diagrammatic view of an apparatus capable of carrying out the process.

Referring more particularly to the drawing, 10 and 11 indicate mixing devices for mixing proper proportions of dry powdered alkali with a saponifiable material; 12 and 13 indicate heating devices for the mixture prepared in the mixers 10 and 11; 14 indicates a vapor separating chamber for separating volatile materials from anhydrous soap produced in the process; 15 and 16 indicate a condenser and receiver, respectively, for the volatile materials, and 16 indicates a vacuum pump for maintaining a vacuum in the vapor separating chamber 14.

The mixing devices 10 and 11 may be provided with agitators 17 driven, for example, by a pulley 18 rotated from any suitable source of power. The mixers 10 and 11 are also preferably provided with heating devices such as a heating jacket 19 through which any suitable heating medium, for example steam, can be circulated by the pipes 20 and 21 so as to maintain the saponifiable material or mixture thereof with the powdered alkali in the flowable condition. The mixing devices 10 and 11 are intended to be used alternatively in preparing mixtures of saponifiable material, for example glyceride oils or higher fatty acid esters of higher fatty alcohols, with anhydrous powdered alkali such as caustic soda. Thus, by closing the valve 22 in the pipe 23 and opening the valve 24 in the pipe 25, such a mixture may be pumped from the mixer 11 by means of the pump 26 while a mixture is being prepared in the mixing device 10.

The pump 26 is utilized to force the mixture through a heating device 12, shown as a coil 27, heated by a burner 28 on gaseous or liquid fuel. To reach the high temperatures contemplated by the present invention, it is usually necessary to employ one or more supplemental heating devices 13 which may be exactly similar to the heating device 12 and include a coil 27 and a burner 28. A pump 28 is preferably associated with each heating device 13 to force the mixture therethrough. Such pumps also have a valuable mixing function by tending to keep the mixture uniform.

The temperature in the heating device 13 is raised sufficiently high to cause reaction between the powdered alkali and the saponifiable material to form soap and liberate any alcohol forming part of the saponifiable material. The temperature in the coil 13 is usually sufficiently high to vaporize all or a substantial part of the alcohol or other vaporizable material and render the soap molten, such that a mixture containing molten soap and vapors is delivered into the vapor separating chamber 14 through the pipe 30. The mixture is preferably discharged against the wall 31 of the vapor separating chamber by nozzles 32 directed toward the wall 31. This wall is preferably maintained in a heated condition by a heating jacket 33 surrounding the vapor separating chamber 14. A suitable heating medium such as, for example, heated mineral oil is circulated through the heating jacket 33 by the pipes 34 and 35 so as to maintain the wall 31 above the melting point of the anhydrous soap produced. This temperature will vary between approximately 450° F. and 620° F., depending upon the type of saponifiable material and alkali employed. The fluid mixture discharged from the nozzles 32 flows down the wall 31 in thin films, thus providing for rapid evaporation of

any liquid volatiles in the anhydrous soap and the rapid liberation of vapors entrained therein. The vaporized material, such as glycerine or higher fatty alcohols, is withdrawn from the vapor separating chamber through a pipe 36 and delivered to the condenser 15 for such vaporizable materials. The condensed materials are collected in the receiver 16, from which they may be periodically or continuously withdrawn through a pipe 37 by any suitable means, such as a pump. A vacuum, which is preferably as high as commercially practicable, for example 27 to 29 inches of mercury, is maintained in the receiver 16, condenser 15 and vapor separating chamber 14 by means of the vacuum pump 16'.

The molten soap from which volatile materials have been removed flows into the housing of a screw conveyor indicated at 38, which conveyor is provided with a cooling jacket 39 for cooling and solidifying the molten soap. The conveyor 38 continuously removes the anhydrous soap from the vapor separating chamber 14 while maintaining a vacuum seal for this chamber, and cools the soap to a temperature at which the soap will not be injured by contact with the atmosphere. The vapor separating chamber 14 and conveyor 38 may be of the type disclosed in the copending application of Benjamin Clayton and Benjamin H. Thurman, Serial No. 119,168, filed January 5, 1937, which application discloses the structure of the vapor separating chamber and conveyor system in detail.

Anhydrous caustic alkalies, for example caustic soda, do not react appreciably with such saponifiable materials as glyceride oils or other esters of higher fatty acids, for example sperm oil, at temperatures below approximately 350° F. Dry anhydrous caustic soda may, therefore, be mixed with such saponifiable materials without appreciable reaction at room temperature or at any temperature necessary to cause the oil to be in a freely flowable condition. It is preferred to preheat the saponifiable material in the mixers 10 and 11 to approximately 140 to 200° F. during mixing of the powdered caustic therewith, so as to cut down the amount of heat necessary to be added subsequently in the process, and to render the mixture easily pumpable.

The proportions of the alkali to saponifiable material should be such as to produce a substantially neutral soap, and will vary with the saponification number of the material being saponified. That is, for an oil having a saponification number of 190, the mixture will consist of one pound of saponifiable material to 0.136 pound of dry anhydrous sodium hydroxide. This produces a mixture in which the disposed solid is only 13.6% of the saponifiable material, and such a mixture can readily be pumped through the heating device 12.

By alternatively supplying the process with materials from one of the mixers and mixing saponifiable material and alkali in the other mixer, the necessary proportions to produce a substantially neutral soap can be easily attained and substantially uniform mixtures prepared and maintained by the agitators 17. The agitators 17 are rotated during the pumping of the material from the mixers 10 and 11 as well as during initial mixing in order to maintain the mixture substantially uniform, and also the pump 26 serves to additionally dispose the alkali in the oil. A colloid mill (not shown) may be employed, if found necessary, but extremely finely powdered anhydrous alkali is available which will ordinarily render the employment of such a colloid mill unnecessary.

Heat is imparted as rapidly as possible to the mixture being forced through the coil 27, and the mixture is maintained in sufficient motion in the heating coil 27 to prevent local overheating thereof. A single heating device may be employed if sufficiently long to provide for reaching the necessary temperature to cause substantially complete saponification. However, it is ordinarily desirable to employ several heating devices 12 and 13 in series with pumps positioned therebetween in order to reduce the pressure necessary to be developed by a single pump forcing the mixture through a heating device or devices of sufficient capacity to reach the desired temperature. Also, the booster pumps, such as pump 29, aid in maintaining the mixture substantially uniform.

Substantial saponification begins in the neighborhood of 350° F., at which time any soap produced is sufficiently plastic to be flowable along with the mixture of saponifiable material and alkali. This is important, as the formation of anhydrous soap at a temperature much below that mentioned would cause a stiffening of the mixture such that extremely high pressures would be necessary to force the same through the heating devices. In order to insure complete saponification of the saponifiable material, it is preferred to raise the temperature of the mixture above the melting point of the alkali employed. Caustic soda melts at 604° F., and it is therefore desirable to reach a temperature of approximately 610 to 625° F. before the resulting mixture of anhydrous soap and volatile materials is delivered into the evaporating chamber when caustic soda is employed. Caustic potash does not melt until a temperature of 716° F. has been reached and temperatures between 716 and 735° F. are within the contemplation of the present invention when employing caustic potash, although employment of caustic soda is preferred in the present process, primarily because of the lower temperatures possible.

Since soap does not begin to form until relatively high temperatures have been reached, the process may be carried on at pressures not substantially in excess of 300 pounds per square inch, and in many cases pressures of 100 to 150 pounds per square inch can be employed. It is preferred to perform the process with as low pressures as practical. It will be appreciated that the pressure will be highest adjacent the entrance to the heating coils 27, and will progressively drop throughout the heating coils and that, therefore, the pressure will be the lowest in the region of highest temperature in any of the coils 27.

It is also preferred to operate the process so that all of the volatile material, such as glycerine or higher fatty alcohols, is in vapor form when discharged into the vapor separating chamber 14, as greater amounts of heat may be imparted to the mixtures in the heating device 13 and no appreciable amount of vaporizable material need be vaporized in the vapor separating chamber 14. However, an appreciable amount of vaporizable material may be delivered into the vapor separating chamber 14 and vaporized therein due to heat added through the wall 31 as well as the contained heat of the materials entering the vapor separating chamber. In order to secure the formation of vapors in at least the last heating device, the discharge pressure thereof should be

relatively low, for example 30 to 100 pounds per square inch.

If the saponifiable material is comprised of esters of higher fatty acids, the glycerine or other alcohol separated from the soap in the vapor separating chamber 14 and condensed in the condenser 15 will be anhydrous and substantially pure. Any free fatty acids present in the saponifiable material will result in the liberation of water during the saponification reaction, thus somewhat diluting the materials condensed in the condenser 15. This is not of material importance with higher fatty acid esters of higher fatty alcohols, as the condensed higher fatty alcohols are immiscible with water and can be easily separated therefrom.

However, when saponifying glycerides the presence of free fatty acids in the saponifiable material will result in a glycerine mixture containing dissolved water, although, with any mixtures of glyceride and fatty acids contemplated in the present invention, the glycerine will be of extremely high concentration, such as 98% or 99%. The presence of free fatty acids in the saponifiable material must, however, not be excessive, as the dry anhydrous caustic reacts with the free fatty acids at relatively low temperatures forming soap and small amounts of water. Thus, large proportions of free fatty acids will cause plastic or solid soap to form before the temperature has become sufficiently high to render the soap flowable. A small percentage of free fatty acids, for example up to 5%, can be present, however, in the saponifiable material.

Also, a relatively small proportion of an inert liquid, for example kerosene, can be added to the material in the mixers 10 and 11 to maintain the mixture flowable until high temperatures have been reached, even if greater amounts of free fatty acids are present. Such liquid will be separated in vapor form in the vapor separating chamber and can later be separated from the condensate collected in the receiver 16.

In the process above described, the so-called "Varrentrapp" reaction must be considered. This reaction is not a pyrolytic decomposition of the soap but is a definite chemical reaction in which caustic alkali reacts with unsaturated fatty acid radicals to liberate hydrogen and form soap containing saturated fatty acid radicals having a lesser number of carbon atoms than the carbon atoms of the original unsaturated fatty acid radicals. The Varrentrapp reaction is a time-consuming reaction, and may be largely avoided in the present process by rapidly raising the temperature above the melting point of the powdered anhydrous caustic alkali and then immediately separating the volatiles from the resulting soap in the vapor separating chamber 14 and quickly cooling the soap thus formed. Since the caustic alkali in the present invention is employed in substantially chemically equivalent quantities with the saponifiable material, an excess of caustic is not present to accelerate the Varrentrapp reaction.

The Varrentrapp reaction is not necessarily undesirable, as it reduces the unsaturation of the fatty acid radicals of the resultant soap, thus increasing the stability of the soap. A harder and better wearing soap is also produced. By varying the amount of time the anhydrous soap mixture is maintained at high temperatures necessary to complete the reaction, the extent to which the Varrentrapp reaction takes place can be varied. Thus soap of varying characteristics can be produced by the present process by prolonging the time of treatment at the high temperatures contemplated. Any hydrogen produced is removed from the system by means of the vacuum pump 16'.

Since the anhydrous mixture of saponifiable material and caustic, and also the resulting mixture of soap and volatile materials, are maintained out of contact with the atmosphere and in motion throughout the entire process, no local overheating or pyrolytic decomposition of the various materials is produced. Any volatile impurities are removed from the soap, thus producing a light colored substantially pure soap from any reasonably pure saponifiable material.

The process finds its chief utility in the saponification of glyceride oils and fats, since substantially pure glycerine in anhydrous form is directly recovered as part of the continuous process of making soap. However, the process also finds utility with other esters of fatty acids, for example, higher fatty acids of higher fatty alcohols such as sperm oil and wool grease.

While I have disclosed the preferred embodiments of this invention, 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 producing soap and recovering glycerine, which comprises, mixing substantially anhydrous powdered alkali with a glyceride oil substantially free of uncombined fatty acids, heating a flowing stream of said mixture to a temperature at least as high as the melting point of said alkali and above the melting point of the soap when anhydrous to produce soap and liberate glycerine, and thereafter delivering said stream into a vapor separating zone and separating said glycerine in substantially anhydrous vapor form from said soap to produce substantially anhydrous molten soap.

2. The process of producing soap and separating volatile materials therefrom, which comprises, mixing a saponifiable material substantially free of uncombined fatty acids with anhydrous caustic alkali, heating a flowing stream of said mixture to a temperature above the melting point of said alkali so as to produce substantially anhydrous molten soap and form vapors of volatile materials contained in said saponifiable materials, delivering said stream into a vapor separating zone and separating said volatile materials in vapor form from said soap in the absence of substantial amounts of water vapor to produce substantially anhydrous molten soap.

3. The process of producing substantially anhydrous soap, which comprises, mixing substantially anhydrous powdered caustic soda with a glycerol ester of a higher fatty acid substantially free of uncombined fatty acids, heating a flowing stream of the resulting mixture out of contact with the atmosphere to a temperature sufficiently high to cause said caustic soda to react with said ester to produce anhydrous molten soap and glycerine vapors, and thereafter delivering said stream into a vapor separating zone, separating glycerine vapors in the absence of substantial quantities of water vapor from said soap under vacuum conditions and condensing said glycerine vapors to assist in maintaining said vacuum and to produce substantially anhydrous liquid glycerine.

4. The process of producing soap, which comprises, mixing a saponifiable material consisting essentially of esters of higher fatty acids substantially free of uncombined fatty acids with

measured proportions of substantially anhydrous powdered caustic alkali, heating a flowing stream of said mixture out of contact with the atmosphere to a temperature above the melting point of said alkali, maintaining sufficient motion of said mixture during said heating to prevent pyrolytic decomposition of the fatty materials in said process, separating volatile materials from said soap by discharging a stream of the same into a vacuum zone, removing vapors substantially free of water vapors from said vacuum zone at a rate sufficient to maintain said vacuum, and removing and cooling said soap before discharging the same into the atmosphere.

5. The process of producing soap, which comprises, mixing substantially anhydrous powdered caustic alkali with a liquid saponifiable material substantially free of uncombined fatty acids and which will not react with said alkali at relatively low temperatures, heating a flowing stream of said mixture while substantially free of water to a temperature sufficiently high to cause said saponifiable material to react with said alkali to form molten soap, and cooling a stream of said soap to a relatively low temperature before discharging the same to the atmosphere.

6. The process of producing soap and separating volatile materials therefrom, which comprises, mixing a saponifiable material containing combined volatile material and substantially free of uncombined fatty acids with anhydrous powdered caustic soda, forming a stream of said mixture, heating said stream to a temperature between approximately 604° F. and 625° F. so as to produce soap and form vapors of said volatile materials substantially free of water vapor, and separating said vapors from said soap.

7. The process of producing soap, which comprises, mixing substantially anhydrous caustic alkali with a saponifiable material substantially free of uncombined fatty acids, and heating a flowing stream of said mixture in the absence of substantial quantities of water to a temperature at least as high as the melting point of said alkali to cause said alkali to react with said saponifiable material to produce substantially anhydrous molten soap.

8. The process of producing soap, which comprises, mixing substantially anhydrous powdered caustic alkali with a saponifiable material substantially free of uncombined fatty acids, heating a flowing stream of said mixture in the absence of substantial quantities of water out of contact with the atmosphere to a temperature at least as high as the melting point of said alkali to cause said alkali to react with said saponifiable material to produce substantially anhydrous molten soap.

9. The process of producing soap, which comprises, mixing substantially anhydrous powdered alkali with a saponifiable material substantially free of uncombined fatty acids, heating a flowing stream of said mixture in the absence of substantial quantities of water and out of contact with the atmosphere to a temperature sufficiently high to cause said powdered alkali to react with said saponifiable material to produce substantially anhydrous molten soap.

10. The process of producing soap, which comprises, mixing substantially anhydrous powdered caustic alkali with a saponifiable material substantially free of uncombined fatty acids, and heating a flowing stream of said mixture in the absence of substantial quantities of water and in the presence of an inert viscosity-reducing liquid comprising kerosene to a temperature sufficiently high to cause said alkali to react with said saponifiable material to produce substantially anhydrous molten soap.

BENJAMIN H. THURMAN.