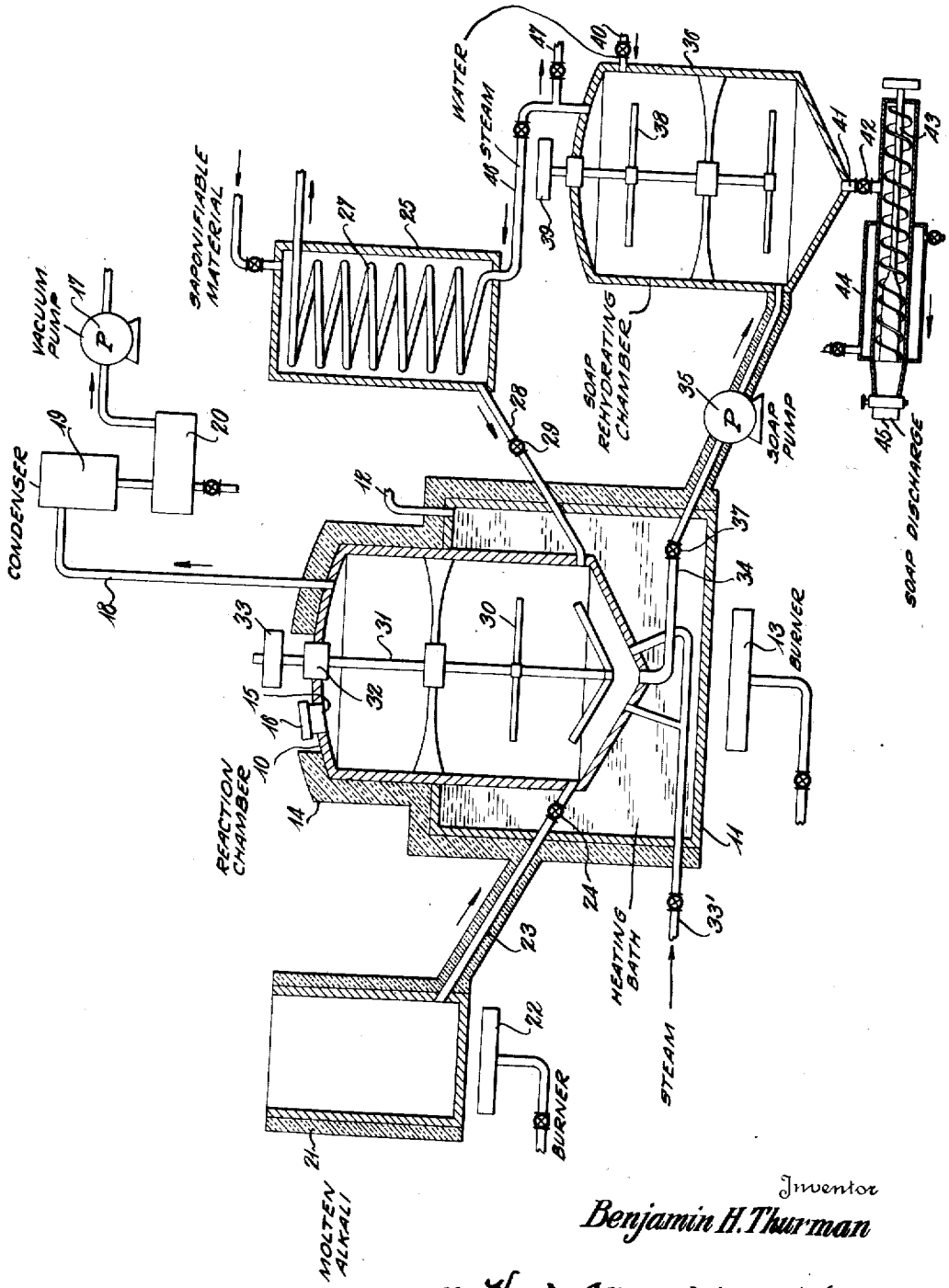
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PROCESS OF MAKING SOAP

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PROCESS OF MAKING SOAP

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4 Claims. (Cl. 260—417)

This invention relates to a process of making soap, and more particularly to a process of reacting saponifiable materials with molten alkalies.

An object of the present invention is to provide a process of reacting saponifiable material with molten anhydrous alkalies in order to produce anhydrous soap.

Another object of the invention is to provide a process of producing anhydrous soap and subsequently cooling and rehydrating said soap to any desired water content.

Another object of the invention is to provide a process of reacting saponifiable materials containing combined volatile materials with molten alkalies so as to produce anhydrous soap and directly recover said volatile materials from said soap.

A further object of the invention is to provide a process of producing soap wherein saponifiable materials are introduced into a body of molten anhydrous caustic alkali, whereby anhydrous soap is produced.

A still further object of the present invention is to provide a process of making soap in which the fatty acid radicals of the resultant soap have a higher degree of saturation than those of the saponifiable material.

In accordance with the present invention, saponifiable material is gradually introduced into a body of molten alkali, preferably below the surface thereof, so as to form a saponified mixture. The process may be carried on as a batch process or a semi-continuous process, and, in either case, the resulting soap may be cooled and hydrated to any desired extent.

Also, in the case of glycerides of fatty acids, or other esters of fatty acids, vaporizable materials such as glycerine or higher fatty alcohols can be directly vaporized from the soap during the formation thereof, and condensed and recovered.

The attached drawing is a diagrammatical illustration of the present process.

Referring to the drawing: 10 indicates a reaction chamber in which the saponification reactions may be carried out. The reaction chamber is provided with a heating means, such, for example, as a chamber 11 surrounding the lower portion of the reaction chamber and adapted to contain a heat transferring medium such as a molten low-temperature-melting alloy or a heated mineral oil. The chamber 11 may be provided with a vent 12 and a heating device such as a burner 13 for gaseous or liquid fuel for heating the material contained in the chamber 11. Since accurate temperature control of the reaction chamber 10 is desired, the structure just described is particularly adaptable, but any other suitable means for heating the reaction chamber can be employed, and also the heating bath contained in the chamber 11 may be heated by any other suitable means, such as by electrical heating elements or any conventional furnace structure. The reaction chamber 10 and heating chamber 11 are preferably surrounded by an insulating cover 14 in order to maintain the temperature substantially uniform throughout the reaction chamber and prevent heat losses.

For batch operation, alkali may be charged directly into the reaction chamber 10 through an opening 15 capable of being sealed by a removable closure member 16. Such alkali can be melted directly in the reaction chamber 10 while the closure member 16 is removed, or any vaporizable material contained in the alkali may be removed during melting by the vacuum pump 17 connected to the reaction chamber through a vapor withdrawal conduit 18, condenser 19 and receiver 20. Even for such batch operation, it is preferred to melt the alkali externally of the reaction chamber in a container 21 arranged to be heated by a burner 22 for gaseous or liquid fuel, or any other type of heating device. The alkali may be melted in the receptacle 21, either in batches or by intermittently adding alkali to a molten mass of alkali in said receptacle 29. The molten alkali may be delivered into the evaporating chamber through a heat insulated pipe 23 provided with a valve 24 which is preferably positioned in the heating bath contained in the chamber 11, in order to prevent solidification of alkali in said valve and consequent difficult operation thereof. The valve 24 may be arranged to be operated by means (not shown) extending externally of the chamber 11.

Saponifiable material may be preheated in a chamber 25 which may be closed to the atmosphere, if desired, although an open chamber may be employed if temperatures below that which will injure the saponifiable material in contact with the atmosphere are employed. Chamber 25 may contain a heating coil 27, or any other type of heating device for heating the material contained in the chamber 25. The saponifiable material may be delivered into the reaction chamber 10 through a pipe 28 provided with a valve 29. An agitator 30 is preferably provided in the reaction chamber 10. The shaft 31 of the agitator may pass through a packing gland 32 and be

rotated from any suitable source of power through the pulley 33. Or for certain operations where anhydrous condensates are not required, superheated steam may be introduced through a pipe 33' to agitate the material in the reaction chamber and assist in vaporization.

The resulting soap may be withdrawn through a pipe 34 by a pump 35, and delivered into a cooling and rehydrating chamber 36. Pipe 34 is provided with a valve 37, also preferably positioned in the heating bath contained in the chamber 11, in the same manner as valve 24.

The cooling and rehydrating chamber 36 is preferably closed to the atmosphere, and provided with an agitator 38 driven from any suitable source of power through a pulley 39. Water in controlled amounts may be introduced into the cooling and rehydrating chamber 36 through a pipe 40, and cooled and rehydrated soap withdrawn from the chamber 36 through a pipe 41 provided with a valve 42. The cooled and rehydrated soap withdrawn, while still at sufficient temperature to maintain the same liquid, is introduced into a conveyor 43 provided with a cooling jacket 44 so that the rehydrated soap may be cooled to solid form and extruded through the discharge opening 45 of the cover.

Large amounts of steam are formed in the chamber 36 during cooling and rehydration of the soap, and at least a portion of this steam may be delivered through the pipe 46 to the heating coils 31 and utilized for preheating saponifiable material, while any steam not necessary for heating the saponifiable material may be exhausted through the pipe 47, or superheated and utilized to agitate the materials in the reaction chamber.

In carrying out the process of the present invention, the process is started by forming a body of molten alkali in the reaction chamber 10. This body of molten alkali may be formed by introducing alkali directly into the reaction chamber through the opening 15 and melting the same within the reaction chamber. The alkali is, however, preferably melted in the receptacle 21 and then introduced into the reaction chamber, as the step of melting the alkali can then be carried on simultaneously with a saponifying reaction within the chamber 10.

After the body of molten alkali has been formed in the chamber 10, the saponifiable material is introduced through the pipe 28, preferably below the surface of the molten alkali, by opening the valve 29. The reaction of the alkali and saponifiable material is extremely rapid, and a substantially uniform mixture is maintained by means of the agitator 30 or a current of superheated steam. Since the saponifiable material is rapidly converted into soap, the saponifiable material is not subjected to extremely high temperatures for any appreciable length of time, and no substantial decomposition of the saponifiable material is produced. If the saponifiable material consists essentially of glycerides of fatty acids or higher fatty alcohol esters of fatty acids, such as sperm oil, glycerine, or higher alcohols, respectively, are liberated, withdrawn through the pipe 18 and condensed in the condenser 19. The vacuum pump 17 is utilized to maintain as high a vacuum as practicable in the evaporating chamber 10, so that the volatile materials are promptly withdrawn and cooled before any appreciable decomposition takes place.

The saponifiable material is gradually introduced into the body of molten material in the reaction chamber 10 until a substantially neutral soap is produced therein. The temperature within the reaction chamber is maintained sufficiently high to keep the resulting soap in molten condition, and, since the reacting mass never becomes acid, decomposition of fatty materials is minimized. Substantially exact neutralization may be obtained by withdrawing samples of the reacting mixture from time to time by test conduits (not shown), testing the same, and then adding sufficient saponifiable material to make the reacting mass neutral.

When the molten mass of soap is substantially neutral, it is withdrawn from the reacting chamber 10 and introduced into the cooling and hydrating chamber 36. Water may be added through the pipe 40, and the mass agitated by means of the agitator 38. Large amounts of steam are formed, and, as stated before, such steam can be utilized to preheat the saponifiable material or agitate the materials in the reaction chamber. When sufficient water has been added to the soap in the chamber 36 to reduce the temperature thereof below the boiling point of water, additional water can be added to bring the soap to any desired degree of hydration. Since commercial bar soap or powdered soap contains between approximately 10% and 30% water, such a commercial soap may be prepared in the chamber 36. The degree of hydration of the soap in the chamber 36 may be determined by withdrawing samples thereof from time to time through test conduits (not shown).

By maintaining the hydrated soap in the chamber 36 at an elevated temperature, for example a temperature in the neighborhood of 212° F. or higher, it can be maintained in the liquid condition and withdrawn through the pipe 41 by opening the valve 42 so as to discharge the soap into the conveyor 43 by which it may be cooled and extruded, as before described. If it is desired to produce a soap having a relatively low degree of hydration, the chamber 36 may be operated under pressure by partially closing the valves shown in pipes 46 and 47, so that a higher temperature than that corresponding to the boiling point of water at atmospheric pressure may be maintained in the chamber 36, in order to maintain the soap in liquid condition.

The process may also be carried on in a semi-continuous manner. After a body of substantially neutral anhydrous molten soap has been formed in the reaction chamber 10, it may be partially withdrawn and additional molten alkali introduced through the pipe 23 into the chamber 10. Additional saponifiable material may be introduced gradually through the pipe 28 until another body of substantially neutral anhydrous molten soap has been formed, which is again withdrawn, and then additional melted alkali added, etc. Thus, additional alkali may be melted during the reaction period in the reaction chamber 10 and at the same time the previous body of molten anhydrous soap may be cooled and rehydrated in the chamber 36. It is preferred to maintain a vacuum at all times in the reaction chamber 10, and, by utilizing the pump 35, molten anhydrous soap may be withdrawn from the reaction chamber while the vacuum is maintained in the reaction chamber.

While any solid alkali capable of being melted without decomposition may be employed as a saponifying reagent, it is preferred to employ substantially anhydrous caustic alkalies. Anhydrous caustic soda is the preferred saponifying reagent,

since it melts at a lower temperature than caustic potash. By employing caustic alkalies, no substantial amount of incondensable gases is liberated therefrom in the evaporating chamber, as would be the case if carbonates were employed. By employing anhydrous caustic alkalies as the saponifying agent and glycerides as the saponifiable material, substantially anhydrous glycerine vapors are liberated and can be withdrawn through the pipe 18 and condensed in the condenser 19 to produce substantially pure anhydrous glycerine. A similar operation can be carried on with higher fatty alcohol esters of fatty acids, such as sperm oil, and dry higher alcohols recovered in the condenser 19. When employing fatty acids or other saponifiable acids, water vapors will be liberated in the reaction chamber 10, and these can be condensed in the condenser 19 so as to enable the vacuum pump 17 to maintain a relatively high vacuum in the reaction chamber 10.

The temperature in the reaction chamber 10 must be at least as high as the melting point of the alkali employed. When employing anhydrous caustic soda, this temperature must be at least 604° F., while, if anhydrous caustic potash is employed, the temperature must be at least 716° F. Somewhat higher temperatures, ranging up to 625° F. for a caustic soda or 735° F. for caustic potash, are preferably employed in order to insure that the alkali remains in liquid condition. These temperatures are sufficiently high to insure that the resultant soap will be in molten condition, as anhydrous soap is molten at temperatures between approximately 450° F. and 620° F., depending upon the type of saponifiable material employed.

By employing a relatively small reaction chamber, each soap making operation may be carried on in a relatively short period of time so that the soap is not subjected to the high temperatures for extended periods of time. Thus, pyrolytic decomposition of the soap due to heating for long periods of time can be avoided, and also the so-called Varrentrapp reaction can be minimized. In certain instances, it is desirable to carry on the Varrentrapp reaction to a desired extent, and this can be accomplished by maintaining the soap in the reaction chamber for longer periods of time. This reaction is not a pyrolytic decomposition of the fatty material, but is a definite chemical reaction in which a molten caustic alkali reacts with unsaturated fatty acid radicals to liberate hydrogen and produce fatty acid radicals of greater saturation having a lesser number of carbon atoms than the original unsaturated fatty acid radicals. Thus, the Varrentrapp reaction produces a harder and more stable soap.

It is apparent that anhydrous cooled soap may be produced by eliminating the chamber 36 and delivering the molten anhydrous soap from the reaction chamber 10 directly to the cooling conveyor 43. The details of such a conveyor are shown in the copending application to Benjamin Clayton and Benjamin H. Thurman, Serial No. 119,168, filed January 5, 1937.

Thus the present invention provides a process of producing either anhydrous or hydrated soap directly from saponifiable materials and molten alkalies. It also provides for the recovery of volatile materials such as glycerine liberated by the saponification of saponifiable materials containing combined volatiles, and the volatile materials may be recovered in substantially pure liquid anhydrous form. Substantially pure neutral soap is produced, and the character of this soap may be controlled by varying the time the soap remains in the reaction chamber 10 at the high temperatures contemplated.

While the preferred embodiments of this invention have been disclosed, it is to be 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, which comprises, forming a body of molten substantially anhydrous alkali in a closed chamber out of contact with the atmosphere and gradually introducing saponifiable materials into said body of molten alkali until molten substantially anhydrous neutral soap is formed, and cooling the resulting soap before contacting the same with the atmosphere.

2. The process of making soap having a controlled water content, which comprises, forming a body of molten substantially anhydrous alkali, maintaining the said body out of contact with the atmosphere, gradually introducing saponifiable materials into said body while maintaining the temperatures of said body above the melting point of the substantially anhydrous soap produced, continuing the addition of said saponifiable material until a body of substantially neutral molten anhydrous soap is produced, thereafter adding water to said body of molten soap, and withdrawing steam therefrom until the soap is cooled and a definite amount of water remains therein.

3. The process of making soap, which comprises, introducing molten anhydrous caustic alkali into a reaction chamber to form a body of molten anhydrous alkali therein, gradually introducing sufficient saponifiable material into said reaction chamber to cause the same to react with said alkali and produce substantially neutral soap, maintaining a vacuum in said reaction chamber during the introduction of said saponifiable material, maintaining the temperature in said reaction chamber sufficiently high to cause the resultant soap to be in molten anhydrous condition, withdrawing a portion of said anhydrous soap from said reaction chamber, introducing additional anhydrous molten alkali, and successively repeating said process.

4. The process of making soap and recovering substantially pure and anhydrous glycerine, which comprises, forming a body of molten anhydrous caustic alkali in a reaction chamber, producing a vacuum in said chamber, gradually introducing saponifiable materials consisting essentially of glycerides of fatty acids in an amount sufficient to produce substantially neutral soap, heating said reaction chamber during the formation of said soap to a temperature to maintain said soap in substantially anhydrous molten condition and liberate glycerine vapors, withdrawing glycerine vapors from said reaction chamber and condensing the same to form liquid, substantially anhydrous glycerine at a rate sufficient to maintain said vacuum in said reaction chamber.

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