Patented Aug. 28, 1945

2,383,631

UNITED STATES PATENT OFFICE

2,383,631

MANUFACTURING SOAP

Walter Russell Trent, North Arlington, N. J., assignor to Colgate-Palmolive-Peet Company, Jersey City, N. J., a corporation of Delaware

No Drawing. Application March 25, 1942, Serial No. 436,102

3 Claims. (Cl. 260—415)

The present invention relates to processes for making soaps and, more particularly, to processes of preparing soaps from mono-fatty acid esters.

Soaps have been made from time immemorial by saponification of fats and oils. The saponification is generally carried out by intimately mixing such fats and oils with alkaline agents in such proportions as are readily determinable by those skilled in the art. The mixture is heated with agitation, forming soap and glycerine. After completion of the reaction, the soap is salted out, leaving glycerine in solution which is thus separated from the soap. If more of the glycerine is to be removed, successive washings, resulting in considerable aqueous dilution of the glycerine, must be employed. The soap is put in a crutcher, where it is mixed with any desired adjuvant material, and may then be framed or plodded and cut, or run in a plastic condition to steel rolls for flaking. The flakes may be left in this condition or may be ground. The plastic soap may also be forced through a nozzle in a spray tower to form beads or other finely divided particles. Continuous or semi-continuous processes of soap making have been provided, but high temperatures and/or numerous washings are also employed in these for removing glycerine from the soap.

Recently, it has been taught to split fats or oils by hydrolysis with water to obtain free fatty acids, and then to saponify these acids. Free acids react very quickly and vigorously with alkaline agents, as compared with the long process of saponifying fats and oils, but it is difficult to control the reaction and the condition of the product at this speed. Contact of free fatty acids, particularly unsaturated acids, with air, even at moderate temperatures, causes the formation of dark oxidation products which tend to discolor soaps. Furthermore, the use of free acids requires that expensive, corrosion-resistant equipment be employed.

It is an object of the present invention to provide a rapid and economical process for making soaps, wherein lower alkyl mono-fatty acid esters are employed.

It is another object of the invention to provide a novel process of manufacturing soap of controlled water content.

It is also an object of this invention to provide a novel and improved method for the continuous manufacture of soap from fatty acid esters of lower monohydric alcohols.

The invention also contemplates the provision of a novel process for the preparation of soap in granular, globular or other comminuted forms.

A further object of the invention is to provide a novel process for the direct and continuous preparation of superfatted soap of pleasant odor substantially devoid of glycerine and possessing desirable properties.

According to this invention, soap is prepared by mixing a fatty acid ester of a lower monohydric alcohol with an alkaline or saponifying agent, the ensuing reaction being carried out at atmospheric, superatmospheric, or reduced pressures and the monohydric alcohol liberated being flashed therefrom. The materials are preferably reacted at somewhat elevated temperatures and under superatmospheric pressure and then flashed into a lower pressure chamber to volatilize the alcohols, as well as some or all of any solvent (including water) employed. A subatmospheric pressure is advantageously employed in the flash chamber to remove from the product alcohol liberated in the reaction. The reaction is preferably carried out as a continuous process, operating from a continuous method of producing monoesters by alcoholysis of fats and fatty oils, using a measuring or proportioning device for mixing the monoesters with the alkaline agent in such proportions as are determined by the operator, and then continuously passing them to a saponifier, as will be described in more detail infra.

In selecting fatty acid esters for use in the present invention, it is preferred to employ fatty acid esters of short chain monohydric alcohols, especially of alcohols having a boiling point in the presence of excess water of lower than 100° C. at atmospheric pressure, and preferably the lower alcohols having 1 to about 6 carbon atoms to the molecule. The fatty acids are preferably those having about 12 to about 20 carbon atoms, and mixtures of the esters may also be used. Mono-fatty acid esters of polyhydric alcohols may also be present in admixture with monohydric alcohol esters. Esters satisfactory for use in the process of the present invention include ethyl laurate, methyl stearate, ethyl margarate, propyl oleate, ethyl esters of coconut oil fatty acids, isopropyl esters of tallow fatty acids, methyl esters of tall oil fatty acids, butyl palmitate, tertiary butyl laurate, methyl arachidate, amyl myristate, isobutyl esters of cottonseed oil fatty acids, benzyl palmitate, and other monohydric alcohol esters. Among the monoesters of polyhydric alcohols which may be present in admixture with the esters of monohydric alcohols are

ethylene glycol monostearate, propylene glycol monolaurate, trimethylene glycol monoesters of olive oil fatty acids, glyceryl alpha- (or beta-) monostearate, mannitol monoesters of coconut oil fatty acids, sorbitol monopalmitate, erythritol mono-oleate, etc. Any of these monoesters may be mixed with di-, tri-, or poly- esters for treatment in accordance with this invention, although such modification is not preferred.

The alkaline or saponifying agents which may be used for saponifying these esters include sodium and potassium hydroxides, carbonates, silicates, etc., pyridine, methyl morpholine, piperidine, alkyl amines, alkanolamines and other organic and inorganic bases and alkaline materials, and mixtures of these. The alkaline agent may be introduced in aqueous solution, in alcoholic solution, or in solutions of other solvents, or may be substantially anhydrous and/or substantially undiluted. The amount of solvent introduced with the alkaline or saponifying agent has a bearing on the water or organic solvent content of the finished product.

The process of the invention can be carried out by intimately mixing a lower alkyl mono-fatty acid ester with a predetermined amount of an alkaline agent in a reaction vessel. The heat of reaction may alone be sufficient to raise the mixture to the desired temperature, depending upon the materials employed and subsequent operating conditions, or heat from external sources may be supplied. Either or both of the reactants may be preheated by steam or other means before being run into the vessel and/or the mixture may be heated during and/or after admixture. Soap and alcohol are thereby formed, and the alcohol is removed from the soap in a vapor separation chamber.

The operation of this process can be carried out continuously by contacting a monoester with a saponifying agent under conditions of temperature and pressure adapted for saponification of the ester. The temperature of the saponification mixture may be varied over a wide range, depending upon the type of ester to be saponified, the amount of water and/or other solvent present, and the type of product desired. Even at only slightly elevated temperatures, the reaction rates are relatively high, as compared with polyesters, such as fats and oils. The temperature employed is partially controlled by the pressure used. Thus, superatmospheric pressures of up to 10 atmospheres and even higher can be used, although pressures of about 20 to about 60 pounds per square inch absolute are preferred. In general, the elevated temperature at which the saponification is carried out may vary from about 20° to about 200° C., although temperatures of about 50° to about 125° C. and pressures of about 20 to about 30 pounds per square inch absolute are preferred. Higher pressures can be employed but in most cases are unnecessary, frequently contributing difficulties of operation and sometimes leading to higher temperatures, which may have a tendency, particularly with unsaturated esters, to darken the products. When operating at atmospheric pressure or below, temperatures of up to about 100° C. can be used. At these temperatures, products of very light color are obtainable. Moreover, the reaction may be carried out in the presence of air, as the neutral esters are substantially undarkened by this treatment.

Soap can also be made by this process by reacting materials under vacuum conditions and flashing into a zone at still lower pressures. The monoester and alkali, preferably preheated to a desired temperature, are automatically proportioned and delivered, preferably continuously, to a mixing vessel where the ester is saponified under reduced pressure. The mixing vessel may be supplied with heat transfer means, such as a jacket, for heating or cooling, as required, and the solvent for the alkali, as well as the alcohol liberated by the reaction, may be partially vaporized under the existing pressure conditions, whether subatmospheric, atmospheric or superatmospheric. Regulation of the temperature of the reaction mass is a factor in determining the amount of solvent and/or alcohol volatilized. After flashing, the soap, dried or partially dried, can be removed from the vapor separation or flash vessel and collected.

An advantageous method of operation is to mix the reactants in a mixing vessel under atmospheric pressure and then to pass the products of the reaction to a separation chamber under vacuum. The materials may be reacted in a continuous mixer, such as a plodder, having its discharge under vacuum, and the comminuted product therefrom may be passed through another continuous mixer or plodder to insure complete reaction and to discharge the product into the atmosphere in desired form, such as bars, fibers, tubes, or grains.

The monoesters and saponifying agents may also be reacted at an elevated temperature and under pressure and then flashed into a separation chamber at a lower pressure, whether superatmospheric, atmospheric or reduced, thereby vaporizing the solvent, if used, and the alcohol formed in the reaction. A suitable condensation system can be provided to recover the vaporized materials. In vaporizing the hydroxylated products of reaction, the saponification of the monoesters affords great advantage over saponification of the glycerides or other natural esters, since the alchols liberated can be vaporized for recovery at much lower temperatures and with far less difficulty than is possible with glycerine. Where the reactants employed are nearly anhydrous, considerably lower temperatures can be employed for removal of solvent than are possible with soap made from free fatty acids, as the lower alcohols, including methyl, ethyl, propyl and isopropyl alcohols, can be vaporized below the temperature required at a given pressure for the vaporization of either water or glycerine. Since the lower alcohol liberated is lower-boiling in the presence of sufficient water than glycerine and/or water and requires less heat to volatilize, lower temperatures may be used, and lighter colored products are consequently obtainable.

Another advantage of the present process is that, where the reaction is not carried to completion, there is no rancidity problem arising from the presence of unreacted mono-fatty acid esters in the soap products, as is the case with unreacted fats or fatty acids. A proportion of the mono-fatty acid ester can be left unreacted in the product to provide a superfatting agent therein, and such unreacted residues provide superior, more stable and sweeter-smelling superfatting materials than are the fats and oils disclosed by the prior art. By operating at low temperatures, the excess fatty acid ester can be left in the soap, even though substantially all of the liberated alcohol is volatilized.

The ester and the alkali solution may be forced

under relatively high pressures through a reaction conduit, such as a pipe, while applying heat. The pipe is preferably equipped with a screw conveyor to displace solid material formed when using plastic or viscous mixtures or may be coiled for less viscous mixtures. The materials are thoroughly contacted in the conduit, and, operating preferably continuously, the reaction product can be discharged to a low pressure zone, preferably through a constricted outlet or nozzle. Heat may be supplied to the pipe, if necessary, to raise the temperature of the alcohol and of the water or other solvent in the pipe to the vaporization point at the pressure in said pipe, and the temperature and pressure at any point in the pipe may be kept substantially constant or may be varied in a cyclic manner for the purpose of creating greater turbulence in the reaction conduit by alternately establishing and destroying a vapor phase therein. Upon flashing into the low pressure zone, the heat of the reaction product is employed to vaporize the water or other solvent and the alcohol at the low pressure of the separation chamber, and the vaporized material goes off at the top while the solid soap, having at least a portion of water and other volatile materials removed, falls to the bottom. Various means may be employed for facilitating the reaction of the alkaline agent with the ester. Thus, it has been found convenient to jet the alkaline solution into the liquid ester and to employ a size and shape of reaction pipe which provides a high degree of velocity and turbulence for contacting the materials.

The moisture or solvent content of the finished product can be modified by regulating the heat supplied to the reaction mixture by preheating the reactants and/or externally heating the conduit or otherwise. The temperature required is far below that necessary for glycerine recovery in saponifying glycerides. Therefore, although possible, it is unnecessary and less desirable to go to the temperature of molten anhydrous soap or higher in order to vaporize the monohydric alcohols liberated, as they or their aqueous azeotropes are lower boiling than water. Furthermore, it is possible to recover the soap in hydrated form, but free from the alcohol, without additional hydrating equipment, as required with anhydrous soap obtained in certain methods of saponifying glycerides. Moreover, shorter periods of heating, with consequently diminished danger of local overheating and decomposition, can be employed. Where a dry soap substantially free of alcohol and solvent is desired, alcohol and water and/or solvent are preferably at least partially vaporized before the soap formed enters the low pressure zone. An increase in heat supplied causes more solvent and alcohol to vaporize and thus increases the solvent vapor and/or alcohol vapor in the conduit. This increases the pressure and hence the temperature, which is the temperature of the saturated vapor at the higher pressure. A screw conveyor can be advantageously employed for advancing solid soap in the reaction conduit. For improved results in obtaining, for example, a very dry, alcohol-free soap (say, of less than about 2% moisture or alcohol content), the soap may be treated with superheated steam either during discharge into the flash chamber or afterwards, as will be described infra.

Soap builders, inert materials, anti-oxidants, etc., may be added to the monoester and/or to the saponifying agent before contacting. Since the washing steps, in general use in prior art practice, need not be employed, these modifying agents, even if water-soluble, are found in the product of the present process. Conversely, it is also possible to employ, if desired, volatile, water-insoluble materials, such as low-boiling petroleum hydrocarbons, the presence of which in the final product might be disadvantageous for certain uses, since they do not appear in the finished product. Certain saponification accelerators, while not ordinarily required for saponifying monohydric alcohol esters, fall in this category and may be added, if desired.

The vaporized water or other solvent and the vaporized alcohol liberated in the reaction are withdrawn at the top of the separation chamber and together pass to a condensation system. The temperature of the chamber and of the passage to the condensers is maintained above the dew point of the vapors. The vapors may be conjointly condensed, but it is preferred to provide at least one condenser for each material to be condensed or to employ a fractionating column. The condensers may be of reflux, jet or other suitable type. Recovery of solvent and/or alcohol in relatively pure condition is facilitated by this means.

The pressure within the separation chamber is easily regulated by control of the condenser temperatures and of the pressure beyond the condensers. The pressure within the chamber is preferably low enough to provide rapid vaporization of the highest boiling material to be withdrawn as a vapor. This depends in part upon the temperature and degree of vaporization of the reaction mixture discharged into the chamber from the nozzle, as the higher this temperature and pre-volatilization, the higher can be the pressure within the chamber.

By contacting the reaction mixture, momentarily and just before discharge into the low pressure or flash zone, with an inert gas at a considerably higher temperature, less heat need be supplied to the reaction conduit and/or a higher absolute pressure may be employed in the separation chamber. The contact of the reaction mixture with an inert gas, such as superheated steam, kerosene vapor, carbon dioxide, flue gases, stack gases, etc., is preferably made at extremely high velocity and for a minimum time period before introduction into the flash chamber. The contacting can advantageously be accomplished by a nozzle designed to permit intimate but only momentary direct contact and a minimum of indirect contact before emission into the flash chamber. Employing this modification, the length of the reaction and/or heating conduits can be appreciably shortened, if desired, and less heat need be supplied thereto by external heating of the conduit and/or by pre-heating of the monoester and/or the saponifying agent.

In another procedure, it is possible, by means of superheated steam or other inert gas, to supply sufficient heat at an optimum temperature and velocity in order to effect instantaneous saponification, even under anhydrous conditions, along with flash distillation of the materials to be withdrawn as a vapor. This procedure avoids plugging of pipes and nozzles, because the reactants are not mixed together for a sufficient time before flashing for saponification, especially under anhydrous conditions, to take place to any substantial extent and because the mixed reactants are not excessively heated before introduction into the nozzle, where they attain high ve-

locity and adequate heat in momentary time before their discharge into the flash chamber. Although the flash chamber may be operated in any case at atmospheric, elevated or reduced pressure, depending upon the vapors to be withdrawn and the temperature of the discharge from the reaction conduit, it will be understood from the foregoing that the use of superheated steam or other inert gas at a very high temperature raises the pressure at which the flash chamber can be operated.

Operation according to this latter procedure is simple in that only a few process steps and a small amount of equipment are necessary, and, furthermore, such operation utilizes the chemical heat of saponification. Great economy of operation is achieved by thus getting additional heat from the saponification after contact with the superheated steam and adsorption therefrom, instead of first saponifying the monoester and then heating the soap before contact with the steam.

In practice, the proportion of superheated steam introduced into the nozzle in these various modifications can be so regulated that a dry product or a hydrated product can be produced. The product may be made in a more or less finely divided condition, which permits its ready transformation into flakes or other physical forms, for example, by passing between a pair of properly spaced rolls, or by pressing into cakes, or by other means, with or without addition agents.

Adjuvant materials may be admixed with the soaps thus formed by mixing them with the monoesters and/or the saponifying agents before flashing, by simultaneously flashing a second solution containing such adjuvant materials, and/or by mixing the final product therewith. Such adjuvant materials may include fatty acid soaps prepared by the same or other methods, resin acid soaps, naphthenic and alkylated naphthenic acid soaps, sulphated and sulphonated organic compounds, alkaline soap builders, water-soluble, water-softening compounds of the acids of phosphorus, and other salts including sodium carbonate, sodium silicates, trisodium phosphate, borax, sodium tetraphosphate, sodium bicarbonate, sodium sulphate, sodium chloride, sodium acetate, sodium hypochlorite, sodium thiosulphate, sodium perborate, sodium tartrate, sodium citrate and sodium oxalate, and the corresponding ammonium, substituted ammonium and potassium salts of the corresponding acids; insecticidal, germicidal, styptic and medicinal agents, including aluminum chloride, mercuric chloride and various copper and lead salts; coloring agents, abrasives, fillers, and water-dispersible gums, including dyes, lakes, pigments, silica, kieselguhr, silica gel, feldspar, precipitated chalk, pumice, infusorial earth, bentonite, talc, starch, Irish moss, sugar, methyl cellulose, agar, gum tragacanth, gum arabic, and polyvinyl alcohol; liquids, such as ethyl alcohol, glycerol, cyclohexanol, naphtha, benzene, kerosene, turpentine, pine oil, decalin and tetralin, and the like. The type of addition agent will depend upon the ultimate use of the new composition.

The reaction product from the vapor separation chamber may be discharged into a cooling element or conduit, where it is partially cooled while under pressure, thus partly solidifying the soap. It may then be passed to an extrusion orifice, where it may be cut into bars. During the cooling operation, but while the soap product is still quite liquid due to the presence of water and/or other solvents, any desired modifiers or adjuvant materials, as aforesaid, including builders, abrasives, perfumes, coloring principles, germicides, liquids, air or other gas, etc., may be injected into the soap.

In practice, when producing bars and cakes of soap, it is preferred so to control the cooling of the soap in the cooling element that the soap is at least partially liquid upon passing to the extrusion element. This element, having an orifice of cross-section approximating that of the desired soap cake, is preferably of extended length and jacketed for carrying a cooling fluid for further cooling of the soap. The cooling of the soap may thus be substantially completed in the extrusion orifice during its passage therethrough.

In another but not preferred modification, the soap may by hydrated by steam in the following manner. The mixture from the saponification conduit is ejected through a nozzle into a separation chamber under vacuum under suitable conditions, as disclosed supra, to flash off substantially all water and/or other solvent and substantially all alcohol therein whilst maintaining the temperature sufficiently high for the soap to be in liquid, plastic or semi-plastic state. The soap, substantially anhydrous and non-alcoholic, is then passed to a cooling element, preferably a coil, and steam is injected into the soap during its passage either into or through the cooling element. The soap is then passed to the extrusion element as aforesaid. This provides a relatively simple method for obtaining a desired moisture content of product, as the proportion between the amount of water fed to the boiler for conversion into steam to the amount of monoester fed to the saponification conduit determines the percentage of moisture.

Where a fatty acid ester of an alcohol having a boiling point above that of water and/or where a solvent for the saponifying agent having a boiling point above that of water is employed in the material under treatment, it will be understood from the foregoing that at least part of such alcohol and/or solvent can be retained, if desired, in a substantially anhydrous soap. The temperature (or vacuum) in the flash chamber, for example, can be lowered enough so that all of such alcohol and/or solvent is not vaporized but a portion falls out with the soap. The temperature selected is higher than the boiling point of water at the particular pressure in the chamber but lower than the boiling points of the alcohol and/or solvent. Similarly, where the fatty acid ester of a low-boiling alcohol is used, the temperature and pressure conditions in the flash chamber can be regulated so as to remove substantially all of the liberated low-boiling alcohol, while retaining at least part of the water or other solvent.

If desired, the soap, instead of being forced through an extrusion outlet after flashing, may be ejected through a spray nozzle or spinner bowl into a spray tower. Due to the lower pressure in the tower, the alcohol vapor, steam and air within the soap expand to give beads, and spongy granules. Cooling of the mixture from the vapor separation chamber, as in the manner described, before discharging it into the spray tower may be used to control the amount of alcohol vapor or of steam or solvent vapor produced and hence the amount of alcohol, water or solvent permitted to remain in the soap. A floating granular soap may be made by this treatment by injecting compressed air into the soap during its passage

through the cooling element and by controlling the moisture or alcohol content. The soap, though cooled, is delivered to the spinner bowl or spray nozzle in the liquid, plastic or semi-plastic state, largely depending upon the extent of cooling, and at a temperature of about 105° to about 250° F. Upon atomization, the particles come into contact in the tower with a current of inert gas, preferably air. The air current may be heated and is preferably passed through the tower countercurrent to the sprayed material, although concurrent passage of the air is also contemplated. Substantially granular and/or hollow soap particles are obtained.

The soap may be treated with brine after flashing, if desired, and may then, after separation of the brine and/or a nigre, be passed through a secondary heating coil, where the temperature and pressure are raised, and water in the soap is vaporized and removed to the extent desired in a secondary flash or finishing chamber. It will be understood from the foregoing that the brine treatment may be applied to the soap directly after its formation in a batch reaction vessel before flashing, or, in continuous operation, it may be applied after formation of the soap in the saponification conduit, part of the alcohol and solvent being removed with the brine and impurities before reheating and flashing the purified soap.

The soap may be passed to drum dryers to produce flakes and ribbons. Since drying can be accomplished by other means, as set forth herein, chilling rolls may instead be provided. Where it is desired to form a friable soap, sudden chilling has been found to be advantageous. The drier soaps are more friable than those containing considerable amounts of water, and very dry soaps are frequently self-disintegrating, breaking up into small grains upon cooling. Thus, after removal of alcohol and water or other solvent in the flash chamber, the soap at the bottom of the chamber may be withdrawn by means of sealed conveyors preferably operated under subatmospheric pressures. If the soap is discharged into the flash chamber at a high enough temperature, no additional heat need be supplied; otherwise, the chamber may be jacketed for adding sufficient heat to keep the soap in a fluid condition, or superheated steam may be admitted. The conveyors may also be jacketed, and a plurality of jackets may be employed, so that a heating fluid may be passed through the first part of the jacket (to aid in maintaining a sufficiently high temperature within the flash chamber), while the second part carries a cooling fluid to give the soap a sudden chill. Another and/or additional means of suddenly cooling may be employed by injecting water directly into the soap while in the conveyor, preferably at a part of the conveyor non-contiguous to the outlet of the flash chamber.

While the removal of alcohol formed with the soap acts to cool the soap by the evaporation of the alcohol in the vapor separation chamber, additional water or other liquid may be injected into the soap at temperatures above the boiling point of such injected liquid at the pressure in the vapor separation chamber, the vapors being removed as formed so as not to hydrate the soap. For example, water introduced is immediately vaporized at the temperature of the soap, cooling the soap at a low pressure and in the absence of air, and the water vapor may be passed into the flash chamber. If it is desired to hydrate the soap to any degree, additional water may be injected into the soap, either at this point or farther along, controlling the amount injected. The cooling jacket can be employed for condensing vapors before discharge. Even where considerable hydration is desired and sufficient cooling can be obtained by circulating a cooling fluid in the conveyor jacket, it may nevertheless be advantageous to vaporize part of the injected water for the purpose of obtaining uniform distribution of the water added. Such uniformity may also be obtained by agitation in the conveyor. Economy of operation may be achieved by using one of the reactants, preferably the monoester, as the cooling fluid in the jacket. This serves to preheat the reactant before its passage to the saponification conduit.

Steam introduced into the separating or flash chamber can also be employed to add heat and to keep the soap in fluid condition. The introduction of superheated steam makes it possible to operate the flash chamber at a higher pressure. The steam may be discharged into the chamber at any point therein, but it is preferred to have the inlet near or surrounding the soap discharge nozzle from the saponification conduit. It is also possible to attain the desired temperature by steam jacketing the pipe containing the entering soap. The soap discharge inlet may be directed so that the incoming soap impinges upon the wall of the flash chamber, flowing down relatively slowly and thus affording ample opportunity for vaporization of alcohol and water or other solvent. While a nozzle may be employed, if desired, it is not required in this modification, especially if the reacting mixture is moved into a mixing machine, such as a pump, a screw conveyor or an agitator, after passing through the saponification conduit, in order to complete saponification of all saponifiables. A second heating conduit may be used after this mixing to raise the temperature before admission to the vapor separation chamber.

The following examples described herein are merely illustrative of the present invention, and it will be understood that this invention is not limited thereto.

Example I

A mixture of about 240 parts by weight of the methyl esters of coconut oil fatty acids, about 78 parts of the methyl esters of olive oil fatty acids, and about 75 parts of the methyl esters of tallow fatty acids is heated to about 100° C. and pumped into a reaction vessel. About 170 parts of a 42° Bé. solution of sodium hydroxide at about 100° C. are also pumped into the vessel over the same period, the vessel being under a pressure of about 80 pounds per square inch absolute and equipped with an efficient agitator. The mixture is run through the vessel in about 3 to about 5 minutes, and the temperature of the reaction mixture rises to about 140° C. The reaction mass is then discharged into a vapor separation chamber, which is under a vacuum of about 27½ inches of mercury, and the methyl alcohol liberated in the reaction together with part of the water is flashed, being withdrawn to a condensation system. A portion of the soap product, substantially devoid of alcohol and in the form of granules or pellets, is used as such; a second portion is pressed into bars and cakes, and a third portion, being in a plastic condition, is fed directly into a plodder, where perfume and/or other adjuvant materials in desired quantity are added thereto. The soap is extruded from the plodder through an orifice and is cut into cakes.

Example II

About 8 parts per hour by weight of the methyl esters of coconut oil fatty acids at about 45° C. are contacted with about 5 parts per hour of a 20° Bé. aqueous solution of sodium hydroxide at about the same temperature and with an aqueous solution containing about 0.5 part per hour of tetrasodium pyrophosphate in a short coiled conduit designed to provide turbulent mixing during a short time interval, say of a few seconds. The conduit has a constricted or nozzle outlet, providing a pressure of the reaction mixture of about 3 atmospheres, and about 75 parts per hour of superheated steam under a pressure of about 15 atmospheres are injected into the reaction mixture just inside the nozzle. The nozzle is located in a vapor separation chamber operated under a vacuum of about 27 inches of mercury and jacketed to maintain a temperature of about 160° C. Substantially all of the water and the methyl alcohol liberated by the reaction are flashed and removed from the chamber. The soap, substantially anhydrous and non-alcoholic and containing tetrasodium pyrophosphate, is in granular form and is removed by means of a sealed conveyor.

Example III

About 10 parts by weight per hour of the ethyl esters of coconut oil fatty acids mixed with about 20 parts per hour of the ethyl esters of tallow fatty acids are proportioned into a reaction conduit with about 25 parts per hour of a 25° Bé. solution of caustic soda. The conduit is under a pressure of about 40 pounds per square inch and is raised to a temperature of about 150° C. by direct injection of steam from a 150-pound steam line. The conduit is sufficiently long so that the reactants pass through in about twenty minutes. The material in the conduit is discharged into a jacketed vacuum chamber, wherein the ethyl alcohol liberated and most of the water are vaporized and removed. Soap in granular form and containing about 12% moisture is continuously removed from the bottom of the chamber by means of a sealed conveyor.

Although the present invention has been described with reference to particular embodiments and examples, it will be apparent to those skilled in the art that variations and modifications of this invention can be made and that equivalents can be substituted therein without departing from the principles and true spirit of the invention. Such variations and modifications are believed to be within the scope of the present specification and within the purview of the appended claims.

I claim:

1. A process for producing soap which comprises continuously passing a fatty acid ester of a lesser alkyl monohydric alcohol and a saponifying agent through a mixing zone at an elevated temperature and under a superatmospheric pressure, varying the temperature and pressure in said mixing zone alternately to establish and destroy a vapor phase therein, and continuously discharging said reaction mixture into a lower pressure zone.

2. In the process of producing soap comprising continuously passing a fatty acid ester of a lower alkyl monohydric alcohol and a saponifying agent through a mixing zone at an elevated temperature and under a superatmospheric pressure, and discharging the reaction mixture into a lower pressure zone, the improvement which comprises alternately establishing and destroying a vapor phase in said mixing zone.

3. In the process of producing soap comprising continuously passing a fatty acid ester of a lower alkyl monohydric alcohol and a saponifying agent through a mixing zone at an elevated temperature and under a superatmospheric pressure, and discharging the reaction mixture into a lower pressure zone, the improvement which comprises alternately raising and lowering the temperature and pressure within said mixing zone in a cyclic manner.

WALTER RUSSELL TRENT.