

July 31, 1945.

J. J. JACOBS

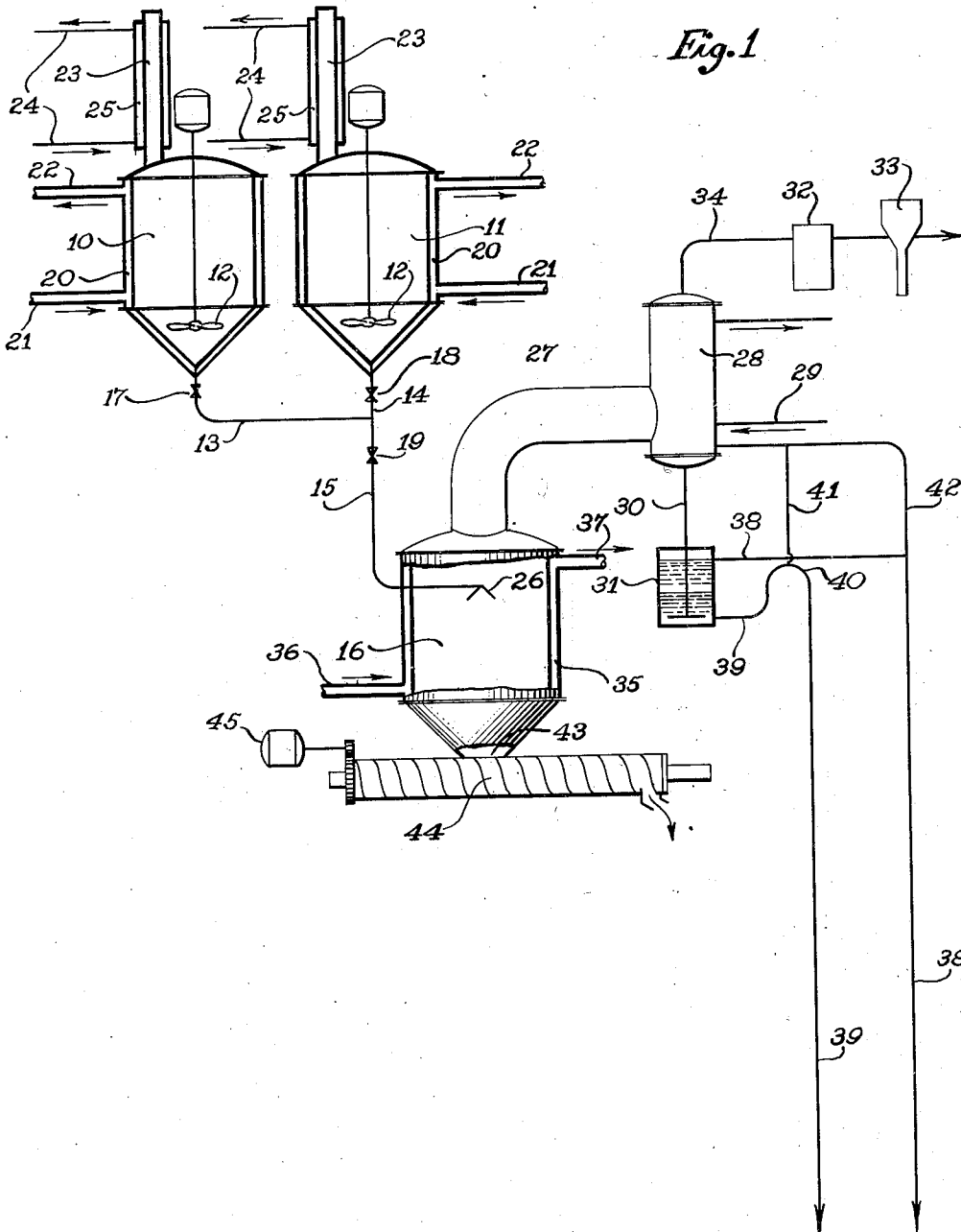
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CONTINUOUS SAPONIFICATION PROCESS

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Fig. 1



INVENTOR
JOSEPH JOHN JACOBS
BY
Frederic P. Worfield
ATTORNEY

July 31, 1945.

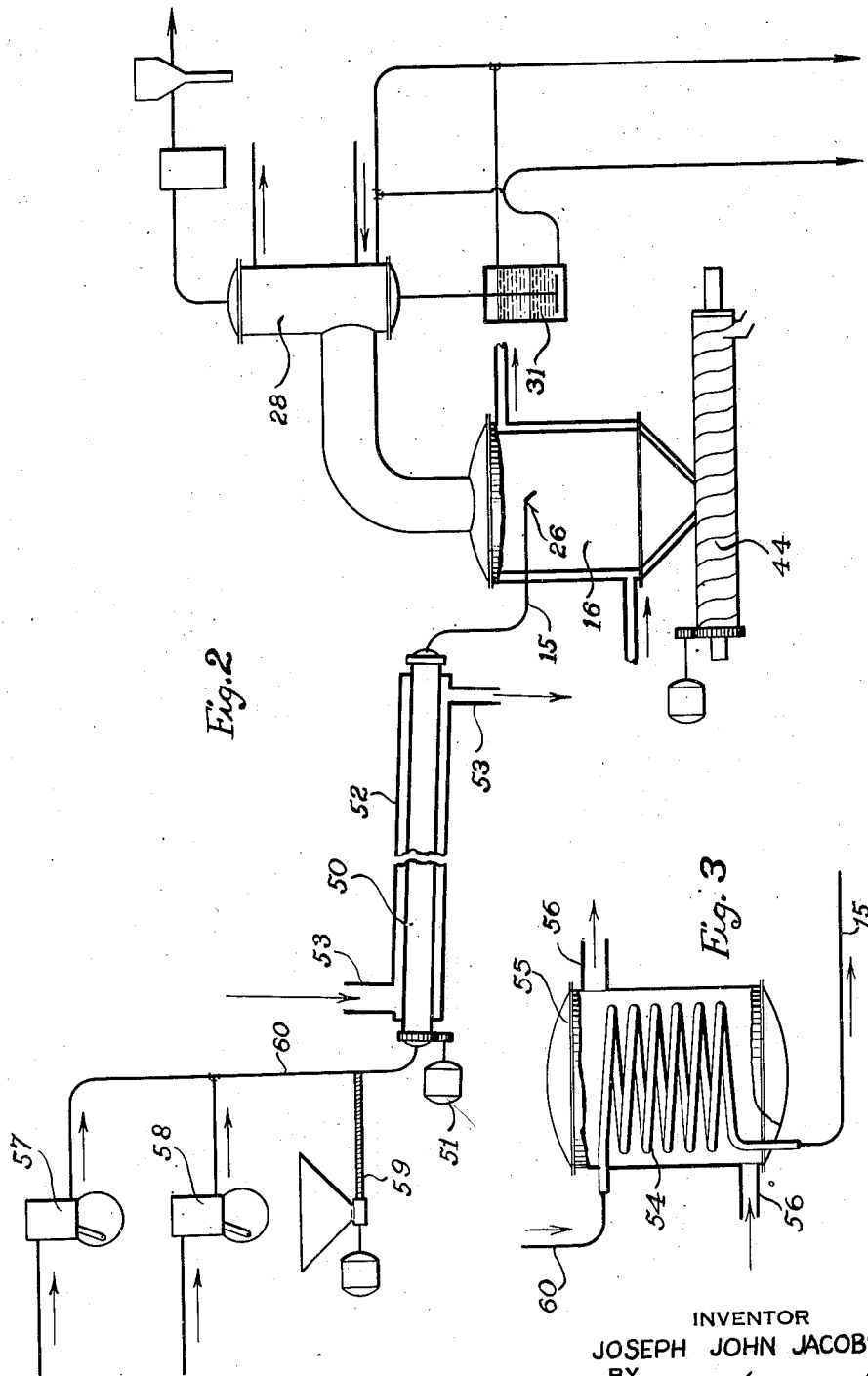
J. J. JACOBS

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INVENTOR
JOSEPH JOHN JACOBS
BY
Fredrick P. Wagoner
ATTORNEY

UNITED STATES PATENT OFFICE

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CONTINUOUS SAPONIFICATION PROCESS

Joseph John Jacobs, New York, N. Y., assignor to
Autoxygen, Inc., New York, N. Y., a corporation
of New York

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This invention relates to saponification and particularly to a continuous saponification process whereby certain desired products of saponification are continuously obtained.

One of the objects of the invention is to provide a continuous saponification process which may be used for manufacturing soap and which is quicker and less expensive than processes heretofore used.

Another object is to provide a continuous process for the manufacture of soap by means of which a desired amount of certain substances, not produced by saponification may be incorporated in the finished product.

Another object of the invention is to provide a continuous process for making naphtha soap, which has a fewer number of steps than processes heretofore used.

Still another object of the invention is to provide an anhydrous soap having a gel structure.

Another object of the invention is to provide a naphtha soap with a larger proportion of naphtha than is contained in naphtha soaps heretofore produced, and which has greater stability than such soaps.

A further object of the invention is to provide a continuous process for manufacturing lubricating grease.

Another object of the invention is to produce a stable anhydrous lubricating grease containing between 60% and 90% soap.

Another object of the invention is to provide a process of manufacturing partial saponification products such as glycerine mono- or di-stearate or mono- or di-oleate.

A further object is to provide an apparatus in which the above processes may be effectively carried out.

The invention accordingly comprises the several steps and the relation of one or more of such steps with respect to each of the others, the apparatus embodying features of construction, combinations and arrangement of parts, adapted to effect such steps, and the products which possess the characteristics, properties, and relation of elements, all as exemplified in the detailed disclosure hereinafter set forth and the scope of the application of which will be indicated in the claims.

For a fuller understanding of the nature and objects of the invention reference should be had to the following detailed description taken in connection with the accompanying drawings, in which:

Fig. 1 is a diagrammatical representation of 55

one form of apparatus for carrying on the process of the invention;

Fig. 2 is a diagrammatical representation of a modified form of the apparatus of Fig. 1; and

Fig. 3 is a diagrammatical representation of an alternative form of part of the apparatus of Fig. 2.

Broadly the invention comprises heating saponifiable material and certain anhydrous alkalis at relatively high temperatures in the presence of a diluent, then suddenly reducing the pressure on the mass to permit substantially instantaneous evaporation of the diluent together with certain volatile products of the saponification process, removing the vapors, and collecting and removing the solid matter thus produced. The evaporation takes place according to the principle of partial pressure distillation which permits evaporation of the diluent and other volatile substances at a lower temperature than would normally be required. The process is made continuous by continuously spraying the hot saponified mixture into an atmosphere of reduced pressure whereby the volatile matter is flashed off and continuously removed, and the solid material is also removed in a continuous manner.

The saponifying reagent may be any alkali used in the soap making industry to produce saponification of fats, oils and waxes. Sodium or potassium hydroxide are most commonly used, and I prefer to use the anhydrous product.

The diluent used in the process may be any substance which is substantially inert to the reactants and the products of saponification, which is immiscible with water and the hydroxy component of the reaction products, which boils at a temperature of less than 300° C. at the discharging pressure and which forms a constant boiling mixture with the volatile constituent or constituents of the products of reaction. Among such materials are kerosene, gas oil, paraffin oil, paraffin wax, high boiling white mineral oils, and diphenyl. Kerosene has been found particularly desirable and is preferred.

The improved saponifying process may best be described in connection with the apparatus shown in the drawings. Referring to Fig. 1 a pair of containers or saponifying kettles 10 and 11 are shown for receiving batches of the soap forming materials. Each of these containers may be provided with an agitator 12, driven by a motor or other suitable means, and the two containers are connected by means of pipes 13 and 14 to a main pipe 15 leading to a spray chamber 16. Pipes 13, 14, and 15 are provided with valves

17, 18, and 19, respectively, to control the flow of material therethrough. The containers or saponifying kettles 10 and 11 are each provided with heating means, which may be in the form of a hollow sheath 20 through which high pressure steam, flue gases, "Dowthern," hot oil, or other heating medium may be passed in the direction of the arrows by means of the inlet and outlet connections 21 and 22. These containers are open to the atmosphere, but are provided with reflux condensers 23 to condense and return any of the material which may vaporize in the saponifying kettles. The reflux condensers are cooled by passing a suitable cooling medium through the pipes 24 and sheaths 25.

The pipe 15 terminates in a spray nozzle 26 within the chamber 16, so that the solution coming out of the nozzle will be very finely divided. The chamber 16 is provided at its upper end with a large outlet pipe 27 which leads directly to a condenser 28 of any suitable type, provided with a cooling means, as, for instance, water circulation pipes 29, so that vapors passing up through the pipe 27 are condensed in the condenser 28. A pipe 30 leads from the lowermost point of the condenser 28 to a container 31 which is provided to collect the distillate. I preferably desire to maintain a vacuum in the chamber 16, and for this purpose I connect the top of the condenser 28 with a surge tank 32 and a steam jet ejector 33 by means of the pipe 34.

In some cases I may wish to heat the spray chamber 16, and for this purpose the chamber is provided with a sheath 35 having inlet and outlet ducts 36 and 37 for leading a suitable heating medium, such as one of those mentioned above, into and out of the sheath.

A pipe 38 is connected to the container 31 at a point about one quarter of the distance down from the top thereof, and this pipe is long enough to provide a barometric leg to balance the reduced pressure within the container 31, so as to draw off any liquid above the level of the connection of the pipe with the container. Another pipe 39 is connected to the bottom of the container 31, and is provided with an upwardly extending loop 40, the uppermost part of which is slightly below the connection of the pipe 38 with the container 31. The uppermost part of the loop 40 is provided with a vent pipe 41 which leads up to the condenser 28, and the pipe 39 is also provided with a vent pipe 42 which leads to the condenser, so that liquid can flow out of the container 31 through the pipe 38 when the level of the liquid reaches the connection of that pipe with the container, and through the pipe 39 when the level of the liquid rises to such a point that the weight is sufficient to balance the column of liquid in the loop 40.

The lower end of the chamber 16 is provided with conical walls, so that solid particles formed in the chamber will move downwardly towards the outlet 43 at the bottom thereof, beneath which is disposed a screw conveyor 44 driven by a suitable motor 45. The arrangement is such that solid matter in the conveyor 44 will act as a seal to maintain the vacuum in the chamber 16.

In the operation of this apparatus for the continuous production of soap, any suitable saponifiable materials may be placed in one of the containers 10 or 11, say the container 10, together with a suitable amount of an anhydrous saponifying agent which may be one of those mentioned above. The valve 17 is closed at this time

to prevent the mixture from flowing out. To this mixture is added the diluent which should be in sufficient quantity to maintain a fluid mass.

The container is then heated to a temperature between 200° C. and 300° C., depending on the boiling point of the diluent used, and the heating is continued for a period of time until the desired amount of saponification has taken place. During this time there is no loss by evaporation of any of the materials in the container since any vapor which is formed therein passes up into the reflux condenser 23 and is condensed and returned to the container.

The amount of the diluent is variable and depends on the particular diluent used and whether or not it is desired to remove all of the glycerine. The amount can be calculated in accordance with the principle of partial pressure distillation so that all or only a portion of the glycerine is removed with the diluent when sprayed into the spray chamber. Kerosene with a boiling range between 220° C. and 280° C. may be used in equal proportions with the fat to remove substantially all of the glycerine. In general, the higher the boiling point of the diluent, the less will be needed.

The diluent, however, has another important function: it serves to transmit heat uniformly to all parts of the mixture during saponification, thus promoting homogeneous reaction and preventing overheating at localized points which might cause charring of the soap. The fat dissolves in the diluent, and every molecule of fat is thus in close proximity with the heat-transmitting diluent.

When sufficient saponification has been produced in the container 10, the valves 17 and 19 are opened, permitting the mixture to flow through the pipe 15 and be sprayed into the chamber 16 from the nozzle 26. The material delivered to the nozzle consists of a fluid gel of soap in the diluent with glycerine in suspension. Since the glycerine and the diluent are mixed together in this fluid gel as it is sprayed from the nozzle, the boiling point of the mixture will be lower than the boiling point of either of the materials. This is because of the principle of partial pressure distillation, the vapor pressure of both materials combining to overcome the external pressure. As each small particle of the liquid leaves the nozzle, therefore, distillation occurs substantially instantaneously, causing the glycerine and diluent to flash into vapor which passes out through the outlet pipe 27 of the chamber 16 and into the condenser 28 where condensation takes place. At the same time the solid soap particles precipitate out and fall to the bottom of the chamber 16 where they pass into the screw conveyor 44, from which they may be delivered to suitable molds or packaging apparatus.

The distillate which collects in the container 31 will be glycerine and the diluent. Since the diluent is lighter than the glycerine it will rise to the top; hence the diluent will be drawn off through the pipe 38, while the glycerine will be drawn off through the pipe 39. Before any liquid will flow through the pipe 39 the head of a column of liquid in the container 31 must be greater than that in the container side of the loop 40, and since this portion of the loop 40 contains heavier glycerine and the container has the lighter diluent above the glycerine, the height of the loop 40 is slightly less than the height of

the liquid in the container 31, as maintained by the level of the outlet thereof through pipe 38. As the liquid continuously collects in the container 31, it is continuously separated into the diluent and the glycerine and the two are continuously drawn off through pipes 38 and 39, the barometric legs being necessary in these pipes to permit the weight of liquid therein to overcome the effect of the vacuum in the condenser 28.

While the spray chamber 16 has been described as being under a vacuum, it may, in some instances, be maintained at atmospheric pressure, or even at super-atmospheric pressure, depending upon conditions in the saponification kettle. If the saponification kettle is operated under pressure, (and it may be so operated, if desired), the spray chamber can be operated at atmospheric or even higher than atmospheric pressure, provided the sensible heat drop between the saponification kettle and the spray chamber is enough to supply the latent heat necessary to vaporize the volatile materials from the soap.

Ordinarily, however, the saponification kettle is operated at atmospheric pressure and the spray chamber under vacuum. The temperature of the spray chamber is maintained at approximately the boiling point of the mixture of the glycerine and the diluent under the given vacuum. Usually this temperature will be maintained from the mixture without additional heat. However, if necessary the chamber 16 can be heated by means of the sheath 35 through which the heating medium is circulated. If there is not enough sensible heat drop available between the saponification kettle and the spray chamber, a temperature higher than the boiling point of the mixture may be maintained in the spray chamber, causing heat to flow from the heating medium to vaporize the mixture of diluent and glycerine from the soap.

The saponifying kettles 10 and 11 are operated alternately. When one is discharging into the spray chamber 16, the other is being filled and heated to carry on the saponifying process, and by the time the first is empty, saponification has been sufficiently carried on in the second, so that the valves for the second may be opened and those of the first closed, to permit the saponified mixture to flow out of the second kettle and the first kettle to be loaded again for repetition of the aforementioned treatment.

With the arrangement described above, solid soap particles entirely free, if desired, from glycerine and the diluent are continuously delivered from the conveyor 44, when there is a proper adjustment of the various steps of the process. At the same time glycerine is continuously removed as a by-product, and the diluent may be recovered for subsequent use. The time required for saponification depends on such factors as temperature, degree of division of the hydroxide material, and intimacy of mixing. Two saponification kettles have been shown and should be sufficient to maintain a continuous spray in the spray chamber. However, it will be understood that more of these kettles may be connected to the system if found necessary.

The soap may be delivered in a dry state or a sufficient temperature may be maintained at the conveyor 44 to keep it in a plastic condition. Such a condition will aid in maintaining the vacuum in the chamber 16.

The vapor outlet pipe 27 should be of sufficient

size to maintain a low vapor velocity and so prevent soap particles from being carried out with the vapor.

Under certain conditions I may desire to eliminate the two saponification kettles 10 and 11 and provide an apparatus by means of which the materials for the process may be fed continuously into the apparatus without the necessity of alternately filling and emptying the saponifying kettles, as in the apparatus of Fig. 1. To this end the apparatus of Fig. 2 may be employed. Here the spray chamber 16, conveyor 44, condenser 28, and container 31, with the associated pipes and connections, are the same as shown in Fig. 1. However, in this case the saponifying materials are fed through a long reaction tube 50 which may be mounted at its ends for rotation and provided with suitable rotating means as for instance an electric motor 51. The tube 50 is preferably mounted at a slight angle to the horizontal so that materials fed into the upper end will gradually pass towards the lower end as the tube is rotated. Means is provided to heat the tube 50, and to this end a casing 52 is shown surrounding the tube 50, and is provided with inlet and outlet connections 53, so that a heating medium, such as one of the materials mentioned above, might be passed through it. The length of the tube 50 and the rate of rotation, as well as the temperature thereof, are adjusted so that the materials fed in the upper end at a predetermined continuous rate will be thoroughly mixed and saponified to the desired amount when they reach the opposite end of the tube, whereupon the fluid gel is fed into the pipe 15 which delivers it to the chamber 16 through the nozzle 26.

In some instances it is not necessary to rotate the reaction tube 50, in which case the motor 51 and driving mechanism may be omitted.

If a longer reaction time is desired, a greater length of travel for the mixture during saponification may be provided by the arrangement shown in Fig. 3, in which the reaction tube 54 is shown coiled within the container 55, the latter being provided with inlet and outlet connections 56 to permit the heating medium to be circulated around the coiled tubing.

The proper functioning of the apparatus of Figs. 2 and 3 will depend on the feeding of measured quantities of the materials into the reaction tubes 50 or 54. Any suitable measuring devices, well known in the art, may be used for this purpose. Motor driven piston pumps 57 and 58 have been indicated for feeding the liquid saponifiable material and the liquid diluent respectively, while a motor driven conveyor 59, feeding from a hopper, has been indicated for delivering a measured quantity of the solid hydroxide. These devices feed directly into the pipe 60 which is connected to the reaction tube of either Fig. 2 or Fig. 3. The speed of the motors may be adjusted to deliver each material at the proper rate.

The saponification process using a diluent with the saponifiable material and the hydroxide has been used before in a single batch process. The soap produced by this known process has a portion of the diluent remaining in contact with it which has to be subsequently removed by washing with a solvent. The present invention eliminates this washing step, the soap being completely free from diluent, if desired. Also, in the prior known batch process, a large amount of the diluent is necessarily provided, because, as partial pressure distillation takes place, the diluent is

removed from the mixture and the mixture becomes continuously more viscous. In order to insure the proper proportion of diluent towards the end of the batch process, it is necessary to provide a large amount at the beginning. The present continuous process of saponification uses less of the diluent, because there is always the same proportion of diluent, saponifiable material, and hydroxide at the point where the distillation takes place, as there is at the beginning. It is only necessary, therefore, to provide sufficient diluent to accomplish the desired purpose at the moment when the liquid leaves the spray nozzle.

However, less of the diluent is required for another reason. While the theoretical ratio of the diluent to glycerine can be calculated for any given temperature, this theoretical ratio is very difficult to attain, especially in industrial practice. The closeness with which this theoretical ratio is approached is dependent upon such factors as rate of distillation, intimacy of contact, and amount of surface exposed for distillation. While the diluent to glycerine ratio can be improved in the batch method (i. e. less diluent per unit of glycerine) by increased agitation, the actual ratio obtained is still much higher than the theoretical. I have found that by spraying this soap gel into the spray chamber, a much better ratio of diluent to glycerine may be secured. This means less heat required to distill over a unit quantity of glycerine. Thus, while the theoretical ratio of diluent to glycerine is more disadvantageous than under atmospheric pressure, I have found that the ratio of diluent to glycerine obtained by spraying into vacuum is better than obtained by a batch distillation under atmospheric pressure.

The proportions of diluent to the soap making materials may be adjusted so that all of the diluent is vaporized in the spray chamber and passes into the condenser and is removed. However, if desired the adjustment of quantities may be such as to leave a portion of the diluent in the precipitated soap which falls to the bottom of the spray chamber 16. Because of this, the process may be used for making special soap, such as naphtha soap, which has a quantity of naphtha in the finished product. In this case naphtha may be used as the diluent, and a sufficient quantity may be used and other conditions may be adjusted so that the soap particles falling to the bottom will have some of the naphtha combined therewith. Stable naphtha soap may be made in this manner without any necessity for the additional step of adding naphtha, which has been necessary heretofore, and with a great deal larger percentage of naphtha than has been considered practicable. With my process as much as 40% of naphtha may be incorporated in the soap.

Soap greases may be made with the process of the invention by adding lubricating oil to the ingredients introduced into the apparatus. After saponification has been completed in the saponification kettles 10 or 11 of Fig. 1, or the tubes 50 of Fig. 2, or 54 of Fig. 3, the soap gel including the lubricating oil is sprayed into the spray chamber 16 whereupon the glycerine and volatile diluent are vaporized and the soap and heavier oil will fall to the bottom as a grease. Greases with from 60% to 90% of soap may be made in this manner.

As has been stated above, when the proper proportion of diluent and soap forming materials are provided, and the temperature and time of

heating is sufficient, complete saponification may be produced before the gel is introduced to the spray chamber. However, I may change the proportions of the reactants to produce partial saponification and then spray the gel into the spray chamber. By this means I may produce partial saponification products, such as glycerine mono-stearate, glycerine di-stearate, glycerine mono-oleate or glycerine di-oleate.

A feature of the invention is the particular gel structure of the soap which is produced by the process. This gel structure gives the soap a fluffy or spongy characteristic and makes it dissolve in water much quicker than soaps produced by other processes. The reactive effect of the soap is therefore speeded up. Because of this gel structure, the naphtha soap made by my process holds more naphtha and holds the naphtha more tenaciously, since each particle of soap absorbs naphtha rather than being merely coated with it. Lubricating grease made by this process is more stable, since the soap holds the lubricating oil more firmly and separation of the two is prevented.

It has been stated that anhydrous alkali is preferred for the saponifying reagent. The presence of water lessens the homogeneous heating effect of the diluent, since water is not miscible with the fat. It also requires more heat to vaporize it and when vaporized with the glycerine, forms a dilute solution of glycerine, so that where glycerine is to be obtained as a by-product of the process, the water must be subsequently separated therefrom. Water-free glycerine may be directly obtained from the present process if an anhydrous reagent is used.

The diluent has been referred to as "substantially inert" to the reactants and the products of reaction. To obtain a pure product with the process operating at maximum efficiency, a completely inert diluent should be used, but under certain conditions some reaction between the diluent and other ingredients may be tolerated.

It will be seen from the above description that I have provided a process for making soap which may be carried on continuously and which provides a soap of superior quality and at the same time permits recovery of the by-products. The process requires simple apparatus which is easily set up and maintained.

Since certain changes may be made in carrying out the above process and in the product, and modifications effected in the apparatus for practicing the principle thereof, without departing from the scope of the invention, it is intended that all matter contained in the above description or shown in the accompanying drawings shall be interpreted as illustrative and not in a limiting sense.

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

1. The continuous process of saponification which comprises the steps of mixing a saponifiable material, a saponifying alkali, and a volatile diluent, heating said mixture to effect saponification, providing sufficient sensible heat to vaporize the diluent and the hydroxy component of the products of saponification at a predetermined pressure, discharging the saponified mass in a finely divided condition in a zone of lower pressure, and flashing off the volatile constituents of the saponified mass, thus precipitating the non-volatile portions.

2. The continuous process of saponification which comprises mixing a saponifiable material, a

saponifying alkali, and a volatile diluent, which is substantially inert to said material and said alkali and to the products of saponification and is immiscible with water and the hydroxy components of the products of saponification, continuously passing the mixture through a heated re-acting zone to effect saponification, providing sufficient sensible heat to vaporize the diluent and the hydroxy component of the products of saponification at a predetermined pressure, and continuously discharging the saponified mass in a finely divided condition in a zone of lower pressure.

3. The continuous process of saponification which comprises mixing a saponifiable material, a saponifying alkali, and a volatile diluent which is substantially inert to said material and said alkali and to the products of saponification and is immiscible with water and the hydroxy components of the products of saponification, continuously passing the mixture through a heated reacting zone to effect saponification, providing sufficient sensible heat to vaporize the diluent and the hydroxy component of the products of saponification at a predetermined pressure, continuously discharging the saponified mass in a finely divided condition in a zone of lower pressure, and flashing off the volatile constituents of the saponified mass, thus precipitating the non-volatile portions.

4. The process of manufacturing soap which comprises the steps of mixing soap making materials and a diluent which is immiscible with water and substantially inert to the said materials and the products of saponification and boils at a temperature less than 300° C. at a predetermined pressure, heating said mixture at a pressure higher than said predetermined pressure so as to produce saponification, continuously spraying said mixture into an atmosphere of said predetermined pressure, whereby vaporization of the glycerine produced by saponification and of the diluent takes place and the soap produced by saponification is solidified, continuously removing the vapors so formed, and continuously collecting and removing the solid soap.

5. The process of manufacturing soap which comprises heating a mixture of soap making materials and kerosene to a temperature sufficient to cause saponification and for a period of time sufficient to produce the desired amount of saponification, spraying the mixture continuously in an atmosphere of reduced pressure and at a temperature sufficient to cause vaporization of the kerosene and the hydroxy component of the products of saponification, removing the vapors produced by the spraying action, and collecting the solid material formed thereby.