UNITED STATES PATENT OFFICE

2,096,188

PROCESS FOR MANUFACTURING SOAP AND FOR RECOVERING UNSAPONIFIABLE IN-GREDIENTS INCLUDING GLYCERINE, ALCOHOLS, GASES, WATER VAPORS, AND OTHER IMPURITIES

Henry W. F. Lorenz, Jersey City, N. J., and Frederick W. Brown, New York, N. Y., assignors, by direct and mesne assignments, of thirty-three and one-third per cent to said Lorenz, thirty-three and one-third per cent to Theodore M. Seward, and thirty-three and one-third per cent to Charles H. Wilson, both of New York, N. Y.

Filed for application Serial No. 374,179, April 15, 1939. This application June 21, 1938, Serial No. 27,672

6 Claims. (Cl. 87—16)

This invention relates to the process and apparatus for manufacturing chemical products, e.g., soap and glycerine from oils and fats, by means of spraying chemically interactive substances into steam.

The object of this invention is to produce a process for bringing about chemical reactions with steam, superheated or saturated, in such a manner that the reaction product, if non-volatile, results as a more or less dry and pulverulent mass, or as a more or less dry liquid. If there are formed both volatile and non-volatile substances, the two are readily separated in the process; the general purpose being to render a chemical process more expeditious and considerably cheaper than when carried out by present methods. Thus saponifiable oils and fats can be saponified, with caustic alkali, primarily into soap and glycerine with superheated steam.

The process and apparatus invented by us can be very well illustrated by the example of producing soap and glycerine, as will be described below: In soap manufacture in the usual way the oils or fats are mixed with an excess of caustic alkali and boiled in an enclosed or open kettle until saponification is complete. The soap lye is salted out and the glycerine extracted and refined. By our process an excess of alkali is not needed, in fact, only the exact amount of alkali required for neutralizing the fatty acids need be used, a neutral soap being obtained in one operation. Again, we obtain a practically pure grade of glycerine, free from caustic alkali, salt, or other inorganic impurities. Also, practically all the glycerine is nicely and smoothly separated from the soap residue.

The invention consists in the process, to be more fully described hereinafter and particularly set forth in the claims. Said invention may have the apparatus modified to suit the nature of the reacting substances.

The process can be carried out in a number of ways and with a variety of apparatus without departing from the spirit of our invention.

Other objectives and aims of the invention, more or less specific than those referred to above, will be in part obvious and in part pointed out in the course of the following description of the various steps of our process, and the relation of one or more of said steps to the others thereof, constituting the invention; and the scope of protection contemplated will be indicated in the appended drawing wherein we have illustrated schematically, a form of apparatus which may be employed to carry out our invention. The figure illustrates in vertical section and in elevation, said apparatus.

The drawing illustrates schematically a preferred form of apparatus which may be employed for carrying out our improved process.

Before proceeding to a detailed description of the parts of the apparatus and the manner of carrying out the process, it may facilitate the disclosure to state that the process contemplates the spraying and heating of the soap-making materials in such a manner that the water condensed from the caustic solution and from the superheated steam, the glycerine liberated from the oil or fat, and the small percentage of the volatile matter of an oily nature, are drawn off and collected. During the operation the proper amount of a current of superheated steam, is maintained.

Referring more particularly to the parts, and describing the process more fully, we may say that one method of carrying out our process is to mix, or emulsify, the oil or fat with a suitable quantity of caustic soda or potash dissolved in an appropriate quantity of water and to spray (preferably as a mist, "nebulizing") this mixture into a reaction chamber through which passes a proper amount of superheated steam, said reaction chamber having an outlet connected with a condenser, or series of condensers, condenser worm or coil (disposed within a condensing chamber in which the condensing operation is conducted in the usual way), and a distillate receptacle or receiver or receivers. The spray of the oil and caustic alkali striking the current of superheated steam in the reaction chamber causes the instant saponification of the oil with (1) the formation of a dry precipitate of soap on the bottom of said chamber, (2) the liberation of glycerine and some volatile oil which pass off with the steam and are condensed in the receiver. Thus, for every one hundred pounds of the fat or oil, approximately thirteen and one-half pounds of caustic soda, or twenty pounds of caustic potash, dissolved in twenty-five to thirty
pounds of water would be required to form the spraying mixture. Obviously, to obtain a high yield of glycerine and to ensure the immediate and complete saponification of the oil or fat the temperature of the superheated steam must be sufficiently high to bring this about. We have found that a temperature of about 250–300 degrees, centigrade, gives excellent results.

If it is desired not to recover all or any of the glycerine, and only to produce soap, a lower temperature—say about 150–250 degrees, centigrade,—can be used while still ensuring saponification. If the quantities of oil and alkali are present in equi-molecular amounts, a practically neutral soap results.

Before the operation is begun, it is preferable to have the oil and alkali in intimate mixture or emulsion. This latter can be done by the usual methods, or by spraying the oil into the alkali or the alkali in the oil, etc.

Again, the oil and alkali solution can be sprayed separately, in proper proportions, into the reaction chamber, in such a way that the two sprays are intimately mixed, preferably at a point of entering the chamber.

While the spraying can also be accomplished with the oil and an indifferent gas, we prefer to use steam, ordinary or superheated, for the purpose. For instance, to prepare soap and glycerine from cottonseed oil we may proceed as follows:

Spray into the four hundred and thirty pounds of cottonseed oil and fifty-eight pounds of caustic soda dissolved in one hundred pounds of water. Spray this mixture with superheated steam into a receptacle or chamber heated to 250–300 degrees, centigrade. By means of superheated steam which is passed into and through said chamber, the latter having an outlet connected with a condensing system for the glycerine, water, and a small quantity of a volatile alcohol (probably cholesterin and phystosterin).

Within the range of temperatures above set forth to which the sprayed material or materials are heated in the current or in the atmosphere of super-heated steam, the resultant soap is, of course, anhydrous, and within the range of temperatures above stated and even at temperatures considerably below said range, the anhydrous soap is in a molten or semi-liquid or even in a liquid condition. All soaps when heated to temperatures ranging from 150° C. to 250° C. are dry.

The melting temperatures of anhydrous soap lie generally between about 200° C. and 300° C. and these melting points are dependent upon the nature of materials employed. The salts of the pure fatty acids have melting points ranging from 220° C. to 270° C., and inasmuch as all commercial soaps contain impurities and mixtures of salts of different fatty acids, the anhydrous soaps have melting points at temperatures below the melting point of that of the highest melting point of the constituent. The impurities always present in soaps also tend to lower the melting points thereof.

The point at which glycerine boils at atmospheric pressure is around 290° C. We therefore preferably employ temperatures high enough to produce a distillation of all of the glycerine formed during the hydrolysis of the fatty acids. Obviously, we can saponify an oil or fat first in an usual way and spray the mixture into the reaction chamber through which is passing superheated steam, and thus distill off the glycerine.

In the distillate, the volatile oil (floating on the surface) can be readily separated from the glycerine-water solution, the glycerine being evaporated down to any consistency required. It is also understood, that through suitable apparatus the glycerine can be evaporated down to proper concentration in one and the same set of apparatus.

An inert salt, such as sodium silicate, borax, sodium perborate, sodium carbonate, trisodium phosphate, can be added to the oil-alkali solution before spraying. Such an addition facilitates the more rapid elimination of the glycerine from the soap formed.

Whenever we speak of oil in this specification we may also substitute fats and other saponifiable matter, also waxes.

Sodium silicate, borax, sodium perborate, sodium carbonate, trisodium phosphate, also other suitable additions in the form of other substances such as waxes, heavy mineral oils, can be either added to the mixture to be sprayed, before spraying, or can be sprayed into the reaction chamber separately. The resultant soap then contains this substance in intimate mixture.

It is understood that in carrying out other reactions (above described more particularly for soap making), by the method of spraying the chemically reactive substances into superheated steam, e.g. a current of superheated steam, the process and apparatus are modified to suit the particular object in view. Two or more chemically reactive substances may be mixed before spraying (or sprayed separately) and dispersed in one another, or by emulsion. Thus calcium acetate and calcium benzoate in admixture may be sprayed with superheated steam into a retort through which superheated steam is passing, resulting in the formation of acetonaphene, a volatile product and calcium carbonate, which is non-volatile.

It is also understood that the temperature at which a reaction is to be carried out can be varied in wide limits to suit the particular case in hand. It may be stated that respecting the apparatus, the reaction chamber may be adjusted with a worm or screw device at its bottom for removing the precipitated matter formed during the operation.

The process can be applied to a large variety of chemical reactions by thus spraying chemically reactive substances into steam, ordinary, or superheated. The steam itself can be the carrier of one or more of the chemically reactive substances, with which the chemicals sprayed react. Again, the sprayed substances need not be liquids, they may be gases or solids, the latter preferably in a finely divided state, or colloidal in nature. Again, they may be mixtures of gases, of gases and liquids, of gases, liquids and solids, solids and gases, etc., etc.

The drawing, which is an elevation, represents one of various methods of carrying out our process. A and B are tanks, C a steam superheater, D a heating device, E a reaction chamber, F a condenser and OG and HH pumps. 1 is a pipe and 2, 3, 4, 5, 6, 7, 10, 11, 12, 13, and 20 are valves, 22 is an oil burner, 24 a spraying device.

In operation, a proper quantity of saponifiable oil is introduced into tank A through pipe 18. Into tank B, a strong caustic alkali solution just sufficient for saponifying the oil in tank A, is introduced through pipe 18. Steam, obtained from any usual source and directed to enter the superheater C by opening valve 2, valve 6 leading to the heating device D and exit.
valves 1 are also opened, as is valve 5 leading to the reaction chamber E. Valves 10 and 11 on pipes 17 and 18 are now opened and the pumps GG' and HH' started. The oil and alkali are now pumped through pipes 18 and 25 through the heater D, valves 28 and 29 being opened and so regulated that equimolecular quantities of oil and alkali can enter through the spraying device 24, projecting into reaction chamber E as shown at 23. The soap formed in chamber E can be removed through the door 21. The volatile matter, i.e., steam, glycerine, and volatile oil, pass from chamber E to condenser F and are collected through the outlet 27. 27 represents the fine extending from the steam superheater C. 25 represents the inlet for the cooling water for condenser F. 25 represents the outlet for same.

It will accordingly be seen that we have provided a process well adapted to attain, among others, all of the ends and objectives above pointed out, in an extremely facile manner; and that we have provided a better process for manufacturing soap and recovering the unsaponifiable products than has obtained in processes hitherto carried out.

As many changes could be made in this construction without departing from the scope of the following claims, it is intended that all matter contained in the above description or shown in the accompanying drawing shall be interpreted as illustrative only and not in a limiting sense.

Having thus described our invention, what we claim as new and desire to secure by Letters Patent is:

1. A process of producing an anhydrous soap and glycerine in a reaction chamber through which continuously flows a stream of super-heated steam, which consists in spraying an unsaponifiable mixture of fat or oil and a caustic alkali solution in commingling relationship in said stream of super-heated steam, thereby effecting immediate and complete saponification of the saponifiable constituents of said fat or oil removal of the glycerine, moisture and other unsaponifiable constituents, and then recovering the anhydrous soap and glycerine.

2. A process of producing an anhydrous soap and glycerine in a reaction chamber through which continuously flows a stream of super-heated steam, which consists in spraying separate streams of saponifiable fat or oil and a caustic alkali solution in commingling relationship in said stream of super-heated steam, thereby effecting immediate and complete saponification of the saponifiable constituent of said fat or oil and removal of the glycerine, moisture and other unsaponifiable constituents, and then recovering the anhydrous soap and glycerine.

3. A process of producing an anhydrous soap and glycerine in a reaction chamber through which continuously flows a stream of super-heated steam, which consists in spraying saponifiable fat or oil and a caustic alkali solution in commingling relationship in said stream of super-heated steam, thereby effecting immediate and complete saponification of the saponifiable constituents of said fat or oil and removal of the glycerine, moisture and other unsaponifiable constituents, and then recovering the anhydrous soap and glycerine.

4. A process of producing an anhydrous soap and glycerine in a reaction chamber through which continuously flows a stream of super-heated steam heated to a temperature ranging from 250° C. to 290° C., which consists in spraying saponifiable fat or oil and a caustic alkali solution in commingling relationship in said stream of super-heated steam, thereby effecting immediate and complete saponification of the saponifiable constituents of said fat or oil and removal of the glycerine, moisture and other unsaponifiable constituents, and then recovering the anhydrous soap and glycerine.

5. A process of producing an anhydrous soap and glycerine in a reaction chamber through which continuously flows a stream of super-heated steam heated to a temperature ranging from 250° C. to 290° C., which consists in spraying separate streams of saponifiable fat or oil and a caustic alkali solution in commingling relationship in said stream of super-heated steam, thereby effecting immediate and complete saponification of the saponifiable constituents of said fat or oil and removal of the glycerine, moisture and other unsaponifiable constituents, and then recovering the anhydrous soap and glycerine.

6. A process of producing an anhydrous soap and glycerine in a reaction chamber through which continuously flows a stream of super-heated steam heated to a temperature ranging from 250° C. to 300° C., which consists in spraying separate streams of saponifiable fat or oil and a caustic alkali solution in commingling relationship in said stream of super-heated steam, thereby effecting immediate and complete saponification of the saponifiable constituents of said fat or oil and removal of the glycerine, moisture and other unsaponifiable constituents, and then recovering the anhydrous soap and glycerine.

HENRY W. F. LORENZ.
FREDERICK W. BROWN.