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V. MILLS

2,159,397

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Filed Feb. 18, 1936

2 Sheets-Sheet 1

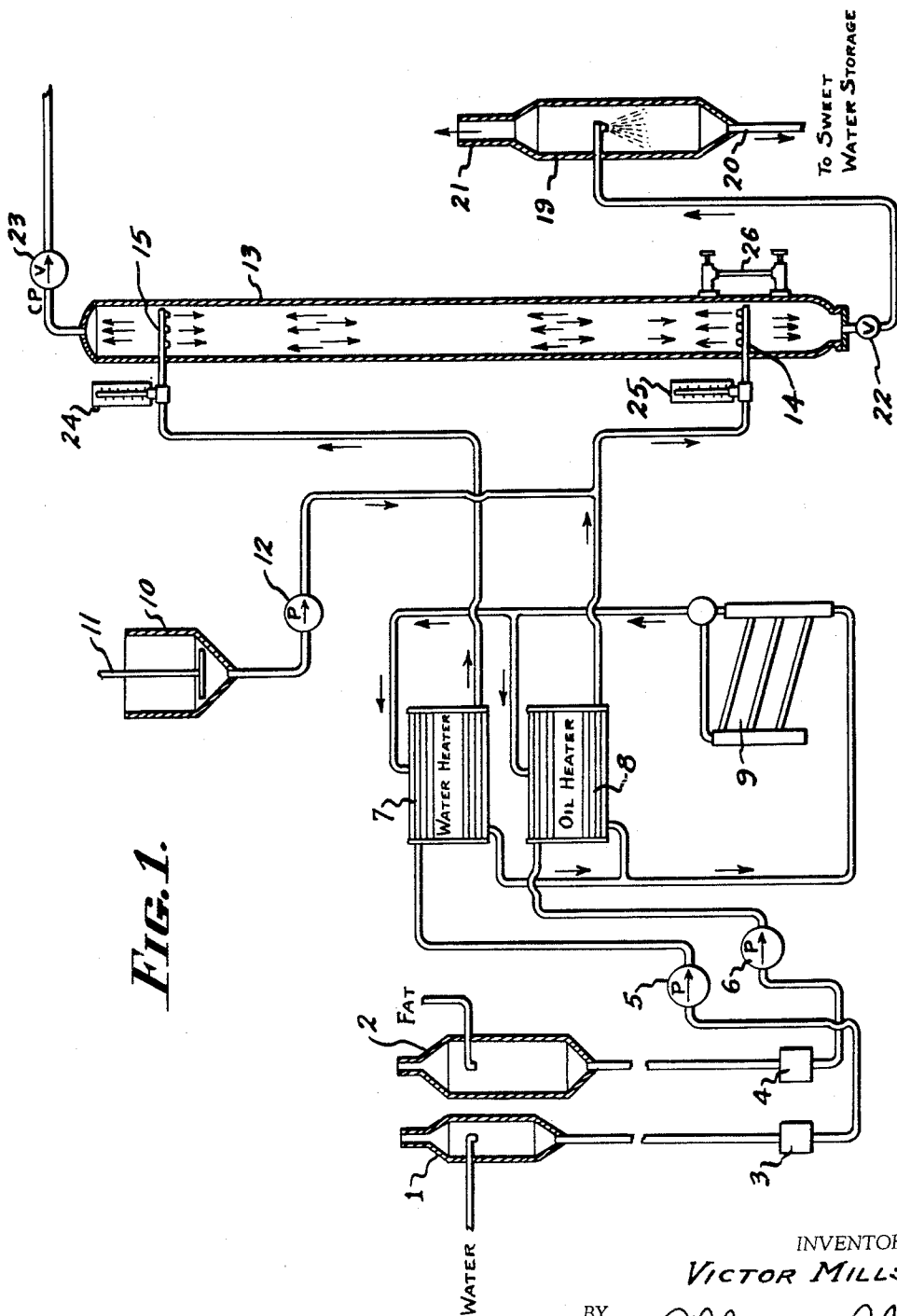


FIG. 1.

INVENTOR.  
VICTOR MILLS.

BY

Allen & Allen

ATTORNEYS.

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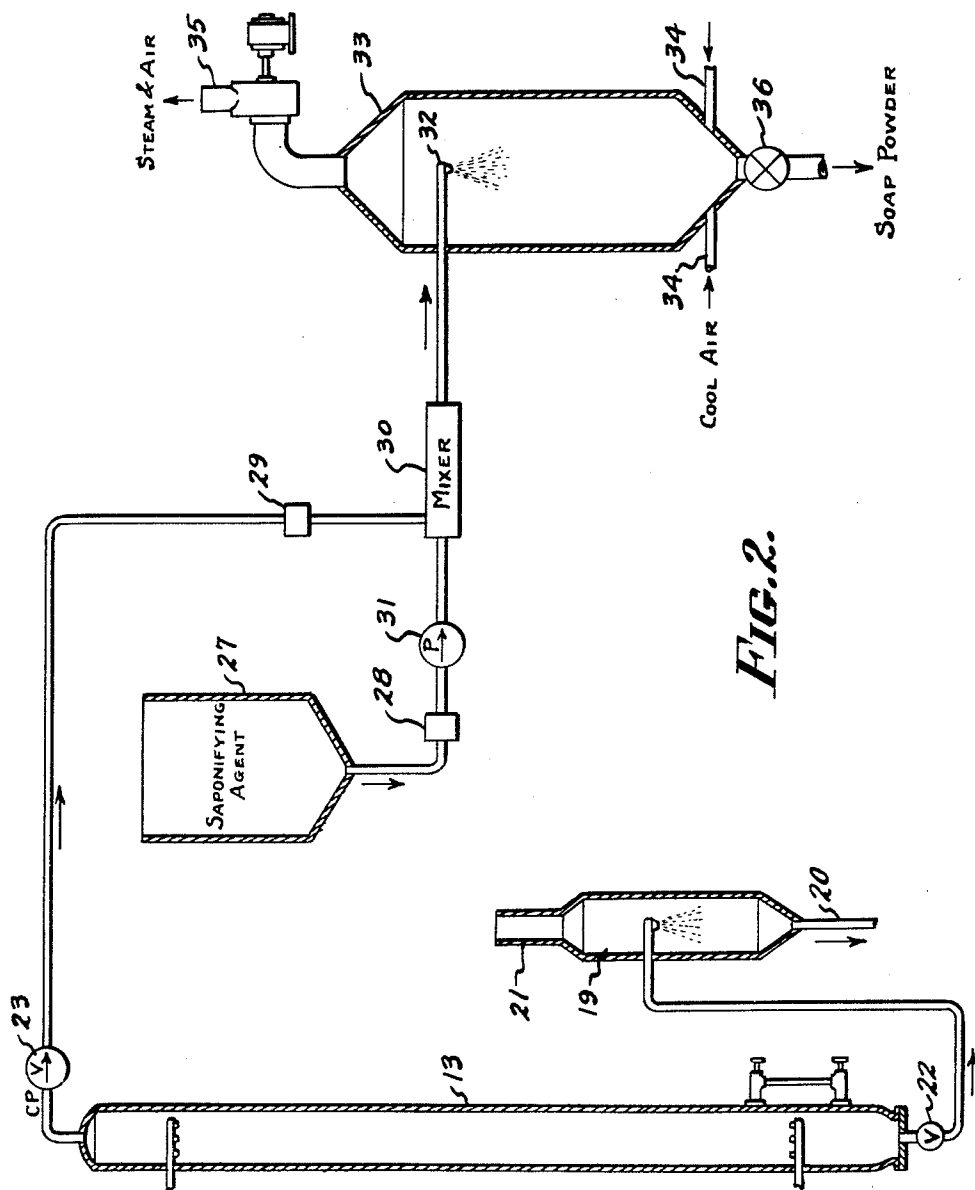


FIG. 2.

INVENTOR.  
VICTOR MILLS.  
BY *Allen & Allen*  
ATTORNEYS.

# UNITED STATES PATENT OFFICE

2,159,397

## CONTINUOUS PROCESS FOR CONVERTING SAPONIFIABLE FATS INTO SOAP AND GLYCERIN

Victor Mills, Cincinnati, Ohio, assignor to The  
Procter & Gamble Company, Cincinnati, Ohio,  
a corporation of Ohio

Application February 18, 1936, Serial No. 64,525

2 Claims. (Cl. 260—415)

My invention relates to a continuous process for converting saponifiable fats into solid comminuted soap and glycerin.

The objects of my invention are to provide a continuous process for the manufacture of comminuted soap and glycerin, including especially the production of a new form of comminuted soap having especially desirable properties, with a great saving in time and space over that hitherto required for accomplishing the same purposes, and a saving in cost of operation.

This application is a continuation-in-part of my co-pending application Serial Number 21,845, entitled Continuous countercurrent hydrolysis of fat.

Soap in comminuted form is becoming increasingly important to the soap maker as its advantages are realized more and more by the consumers. It displaces soap in form of bars and flakes, not only for household laundry use but also for washstand toilet use in dispensers. The forms now marketed range from finely divided, dry, dense particles, slow in dissolving, such as shaving powders, to relatively large sized hollow particles of very light weight, but quick to dissolve. A product that is moderately dense, of rapid dissolving properties, and with moderately high moisture content would have the most desirable combination of properties, and this is accomplished in the present invention, which provides a new and continuous process for making same.

Heretofore the production of any form of comminuted soap has been carried out in a series of separate operations, each of which involves a considerable amount of time and space and a loss of heat due to cooling between operations.

For example, the first step in the usual procedure has been first to convert the fat, (meaning any saponifiable fat or fatty oil), into soap by the usual kettle boiling process which in most large soap factories consumes a period of about ten days for the complete cycle of making finished soap in proper condition for further processing. The glycerin is recovered in this process in the form of a dilute solution containing usually from about five to fifteen per cent of glycerin and considerable salt.

As an alternative procedure in the first step, soap has also been made from the fatty acids derived from saponifiable fats by hydrolysis, either in autoclaves at high temperature and pressure, or by the well known Twitchell process. The fatty acids are saponified with a saponifying agent such as a solution of caustic soda or of soda ash, and converted into the same form of finished soap, all

of which procedure likewise consumes ordinarily several days. Glycerin solution is obtained as one of the products of hydrolysis containing about the same percentage of glycerin as in the aforementioned kettle process of making soap, but substantially free from salts.

In the next step the soap thus made by either procedure may be mixed with suitable builders, etc., if desired, then heated to a suitable temperature for spraying, and sprayed into a chamber where it comes in contact with heated, drying, or cooling air, or other gas, resulting in conversion of the molten soap into various comminuted forms with structure, density, and moisture content varying greatly with the temperature of the soap when sprayed and the temperature and volume of the gas with which it comes in contact in the spraying chamber.

Inasmuch as either of these series of operations consumes several days, large amounts of material must be kept in process at all times to ensure uninterrupted operation; that is, if the complete series of operations requires twelve working days, for example, then the total material in process in the various stages would have to be substantially twelve times the amount of finished product required per day, thus requiring a great deal of space and equipment for a given amount of daily production. The materials at each stage of the processing naturally cool off, and heat must be applied to reheat the material for the next operation.

My process offers a great improvement over these conditions in that it operates continuously; no reheating is required between operations, thus saving the loss and expenditure for fuel and time; and the amount of equipment and space required is extremely small for a given amount of product. The total time consumed in converting saponifiable fat into comminuted soap ready for packaging by my process need not greatly exceed two hours, so the total material in process at any one time would be only that equivalent to two hours' production.

My process consists briefly in first splitting the fat in a continuous autoclave process as described in my co-pending application Serial Number 21,854, then passing the hot fatty acids issuing from same to a mixer where they meet a continuous stream of saponifying agent supplied in proper proportions, such as caustic soda solution for example, whereby saponification occurs almost instantaneously, and then the mixture in the form of fluid soap containing a properly controlled amount of moisture passes without interruption

to a nozzle where it is sprayed into a suitable chamber with a proper supply of air or other cooling or drying gas, resulting in the formation of solid comminuted soap of unique structure and properties, in salable form, with practically any desired moisture content. Any desired builder such as silicate of soda, soda ash, trisodium phosphate, borax, etc., may be added to the solution of saponifying agent before mixing with the fatty acids, or added separately in the mixer, and thus produce a comminuted soap containing such builder. Excessive amounts of crystalline builders, such as soda ash for example, are objectionable because they tend to make a product having a definitely crystalline structure which product tends to break down in handling. I keep the proportion of crystalline builders, when used, below that which produces the above mentioned effects. Hence my mixture as sprayed is a substantially homogeneous fluid mass of soap and water, with or without a builder, and forms a solid product that is also homogeneous, free from noticeable crystalline structure, and resistant to breaking down with ordinary handling.

An important point in making products of good quality by my process is to protect the fatty material and the soap from contact with air or other oxidizing agents from the start until the product is cooled, in order to avoid any darkening effect due to oxidation.

Referring to the accompanying drawings:

Figure 1 shows a diagrammatic elevation view of an apparatus suitable for carrying out the hydrolysis of the fat with resulting formation of fatty acids and glycerin solution, and

Figure 2 represents a diagrammatic elevation view of an apparatus suitable for carrying out my whole process of converting fat into comminuted soap.

Referring to Figure 1, 1 and 2 are deaerators or deoxygenators of any suitable type for the water and fat respectively. 3 and 4 are meters for measuring the two fluids. 5 and 6 are pumps for forcing the water and fat through the apparatus at the desired pressure. 7 and 8 are heaters for water and fat respectively. 9 is a boiler or other device for supplying heating means to the heaters 7 and 8. 10 is a tank for supply of catalyst, in which is an agitator 11. 12 is a pump for delivering the catalyst into the fat. This pump preferably is so arranged as to act proportionally to the fat pump 6. 13 is a vertical autoclave chamber into which the fat is fed at a point near the bottom through distributing pipes 14 and water is fed into the top through distributing pipes indicated by 15. 16 is a tank for receiving the "sweet water", into which it is flashed to atmospheric pressure. The concentrated sweet water then is discharged through pipe 20, and the water vapor through pipe 21. 22 is a valve for suitably regulating the rate of discharge of the "sweet water", and 23 is an automatic pressure control valve on the fatty acid discharge. 24 and 25 are thermometers, respectively in the water and fat inlet pipes to the autoclave 13. 26 is a liquid level gage to show the level of the interface between fat and water in the autoclave. The different parts of the apparatus are suitably connected by pipes as shown and provided with additional valves where needed to properly regulate the flow of the fluids. The autoclave 13 and other parts of the apparatus coming in contact with split fatty acids should preferably be made of a material resistant to the corrosive action of fatty acids. The autoclave

as shown and described herein is about two feet six inches in diameter and fifty feet high, and well insulated externally to reduce losses of heat by radiation. It should be noted that there is a space at the bottom of the autoclave, below fat distributing pipes 14, which will permit the "sweet water" to settle and free itself from globules of fat. The remainder of the autoclave contains fatty matter through which the water is constantly passing. At the top of the chamber or autoclave, above the water distributing pipes 15, is a similar space to permit the fatty acids to separate completely from globules of water before leaving the apparatus.

An exemplary operation of my process consists of the following. In starting up, the water is first passed through the deoxygenator 1, through the meter 3, pump 5 where the pressure is raised to any desired point between 150 and 1600 pounds per square inch, sufficient to maintain the heated water in the liquid phase at the temperature to which it is to be heated, while the pressure in the system is maintained by means of valve 22 (temporarily closed), and automatic pressure control valve 23, on the discharge from the autoclave 13.

The water next passes through the heater 7 where it is heated to the desired temperature, between about 365° F. and about 600° F., and then passes into the autoclave 13 through the distributing pipe 15 until the autoclave is filled with water. The supply of water then ceases temporarily, and pumping of fat begins. The fat such as tallow in a molten state passes through the deoxygenator 2, meter 4, and pump 6 where its pressure is raised to substantially the same point as that of the water. The fat then passes through the heater 8 where its temperature is raised to about that of the water, and then it passes into the autoclave through distributing pipes 14. Any desired amount of catalyst, such as the oxide or soap of zinc, calcium, or magnesium, is introduced into the fat by pump 12, preferably as the fat passes from the heater to the autoclave. The pumping of fat is continued without further addition of water until the autoclave is filled with fat above the level of the water distributing pipes 14, which can be determined by the use of a suitable liquid level gage 26, on autoclave 13. The water dispelled during this period of introducing the fat is allowed to escape through the control valve 22 at the water outlet at the bottom of autoclave 13, at the same rate as that of the fat introduced, while maintaining the desired pressure inside the autoclave to prevent the superheated water from boiling. The apparatus is now in condition for continuous operation.

Both fat and water are now supplied to the autoclave continuously at a steady rate. By supplying tallow at a uniform rate of about 7500 pounds per hour in an apparatus of this size, and water at a rate of about 3500 pounds per hour, while maintaining a temperature of about 470° F. and a pressure of 600 pounds per square inch in the autoclave, the fat takes about one hour to pass through the autoclave, and is hydrolyzed to the extent of over 98 per cent, and usually about 99 per cent. The split fatty acids issue from the top of the autoclave through a suitable pressure controlling valve in a continuous stream. A more rapid passage of the fat and water through the described apparatus in the same relative proportions, taking for example 30 minutes instead of 60 minutes, would still give a high degree of hydrolysis, only a few per cent under that obtained

in 60 minutes. The liberated glycerin is taken up by the water and leaves the bottom of the autoclave in a continuous stream in the form of a solution or "sweet water" which will contain approximately 24 per cent glycerin, if the original fat was of good grade, containing substantially its full complement of glycerin. In order to provide a rather prolonged contact of water with fat, the main body of the autoclave should preferably be kept filled with fat through which the water descends, and the interface between the fat and the accumulated "sweet water" should preferably be maintained in the apparatus described at a point about three feet above the bottom of the autoclave, or at about the level of the fat inlet 14. This level can be regulated by suitably controlling the flow of the fat and the water, and can be determined by use of suitable draw-off valves at various levels or by a liquid level gage.

The "sweet water" passes to the flash tank 17 where its pressure is released through a suitable valve to atmospheric pressure, whereby a portion of the water suddenly evaporates to steam with a resulting cooling effect so that the temperature drops to slightly more than 212° F. with a resulting concentration of the "sweet water" from a glycerin content of 24 per cent to approximately 32 per cent. The split fatty acids issuing from the top of the autoclave may, if desired, be passed to a flash chamber maintained at any desired lower pressure, where the pressure is reduced if desired through a suitable valve, but I prefer to pass the fatty acids without reduction of temperature or pressure directly to the mixer, as described below. The fatty acids while under the temperature and pressure stated, if derived from fats of the tallow class, contain approximately 11 per cent of water in solution, which, however, will partially flash into steam if the pressure is reduced, with a resulting cooling effect on the fatty acids, but ordinarily this entire moisture content is retained in the fatty acids as used in my preferred procedure.

Both the fatty acids and the dissolved water in same are substantially free from glycerin when the operation is carried out as described in an autoclave column of sufficient height. Thus, in a column ten feet high, I find that the water which is dissolved in the issuing fatty acids contains about five per cent of glycerin; in a twenty foot column the water contains only about one per cent glycerin, while in a fifty foot column as described herein the water contains only about 0.1 per cent glycerin.

The fatty acids issuing from the autoclave 13 (containing dissolved water in amount depending on the temperature and pressure), and having a temperature above 212° F., preferably about 470° F., are then passed to mixer 30 through meter 29, while at the same time a solution of a saponifying agent, preferably caustic soda or soda ash in amount to properly saponify the fatty acids, continuously controlled by a suitable proportioning device, is likewise passed continuously into the same mixer by pump 31 from tank 27 through meter 28. Meters 28 and 29 enable the operator to control the flow of saponifying agent and fat in proper proportions as desired, or they may be suitably connected with pump 31 in an automatic proportioning device. When a built soap product is desired, the solution of builder may be added to the solution of saponifying agent, or it may be added to the mixer sepa-

rately in properly and continuously proportioned quantities. The total amount of water introduced in the mixer is so regulated as to give the desired moisture content in the final comminuted soap product, after allowing for evaporation in the spray chamber. The total amount of water in the soap should not be more than sufficient to form the "neat" soap phase, in order to assure ease of handling. No application of heat is necessary in the mixer or subsequently, because the fatty acids as received from the autoclave system already contain an adequate amount of heat for the subsequent operations, having a temperature preferably in the neighborhood of 470° F. The saponifying agent does not need to be heated more than enough to have it in good pumping condition but it may be heated, if desired, to impart additional heat to the soap before spraying to secure maximum evaporation. The mixer must necessarily be kept under a sufficient pressure to prevent volatilization of the water at the temperature of mixing, and should be of sufficient size and capacity to keep the materials in contact with each other long enough to insure intimate contact and completion of the chemical reaction. This period need not ordinarily exceed one minute with an efficient mixer.

The hot soap mixture then passes directly to the spray nozzle 32 and issues therefrom into the spray chamber 33 in comminuted form. Inasmuch as the soap mixture reaches the nozzle at a temperature considerably above 212° F., usually about 250°-400° F., and under super-atmospheric pressure, the pressure drops suddenly to that of the spray chamber, usually atmospheric pressure, with a consequent drop in temperature and instantaneous evaporation of a portion of the water content in the soap. In making my preferred form of product, cooling air is admitted at the bottom of the spray chamber through openings 34 in order to cool the product sufficiently to keep the particles from sticking together, and passes outward at the top through exhaustor 35, thus incidentally carrying away the water vapor volatilized from the soap mixture. The soap mostly falls to the bottom of the chamber where it can be continuously removed through air lock 36 for packaging, or for further processing into other forms if desired. Any fine material which may pass out of the top of the chamber with the air is caught in a suitable dust collector.

Other types of spray dried soap may of course be produced if desired, such as the well known Lamont product described in U. S. Patent 1,652,900, by suitable modifications of the procedure in the spray drying chamber.

The air supplied to the spray chamber may be introduced in either a concurrent or counter-current direction with the flow of soap, and by suitable adjustments of the temperature of air, temperature of soap, and volume of air, practically any desired variation in moisture content, or size, of particles may be obtained.

The preferred soap product obtained as described is novel as far as I know in consisting of feather-like, irregular shaped solid, not hollow, particles of size easily visible to the naked eye, substantially free from glycerin, and may contain practically any desired amount of moisture, percentages between 15 and 25 being especially desirable and readily obtainable, but ten per cent is approximately the lowest percentage readily obtainable in practical operation. A

product with lower moisture constant may, however, be obtained by using molten caustic soda instead of an aqueous solution of same for saponifying. The particles mainly sink in water, but form a bulk product having a bulk density usually of about .25 to .40 as compared with water, due to irregular structure of the individual particles which prevents the particles from packing tightly together, very readily soluble, and fairly free flowing, non-dusting, and shows little or no tendency to lump or ball together in water. In these latter respects it compares favorably with spray dried products of the rounded, puffed, thin-wall type, such as those described in the patent to Lamont No. 1,652,900, which are noted for their ready solubility and their free flowing qualities, but which are light bulking and usually of low moisture content. My product as described dissolves in 10 to 15 seconds in the solubility test described in Lamont's Patent No. 1,652,900, as follows:

" . . . These figures on speed of solubility were determined by placing  $\frac{3}{4}$  grm. of soap product in  $\frac{1}{2}$  liter of water at 110° F. and stirring the product with a tablespoon at the rate of 25 double strokes per quarter minute. The container holding the soap and water was provided with an internal removable screen which was lifted out of the water at desired intervals, say 10 to 15 seconds, and if any soap remained undissolved it could be seen on the screen."

I attribute the rapid solubility of the particles of my preferred product, in spite of their solid interior, to the fact that the particles are thin, have a large irregular surface due to the bursting or explosive effect in their formation, and to the fact that they have not acquired a hard dried skin on the surface of the particles, such as would result from exposure to a highly heated drying gas. All the heat for drying my product is stored up within the product under high temperature and pressure conditions before reaching the spray nozzle, and on issuing from the nozzle at a lower pressure there results a bursting or disintegration of the particle due to the instantaneous volatilization of moisture contained in same.

The glycerin solution continuously discharged from the autoclave in my process has a concentration of approximately fifteen to forty per cent, usually about twenty-four per cent under the preferred conditions described, and is substantially free from salts. This may be further concentrated by "flashing" or suddenly subjecting it to a lower temperature and pressure, in which case the concentration may be raised from twenty-four per cent to as high as thirty-two per cent glycerin. This is much higher than is obtainable in the commercial operation of other soap making and glycerin recovery processes. This solution is then subjected to the ordinary purification, evaporation, and distillation proc-

esses to obtain the pure glycerin of commerce. The recovery cost is obviously much lower than in prior known processes because of the higher initial concentration of the glycerin solution and absence of dissolved salts.

The comminuted soap thus obtained in this process can not only be used and sold as such, but it may also be readily converted into other forms of soap products such as flakes, bars, or extruded forms. For this purpose it is only necessary to produce first the comminuted soap having the formula and moisture content desired, and then pass it through rolls to convert it into flakes, or through suitable rolls and pladders or other known equipment to convert the particles into the form of extruded bars.

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

1. That method of preparation of soap, which consists in continuously forming fatty acids from fats at high temperature and pressure by hydrolysis using an excess of water over that required for hydrolysis and by control of temperature during hydrolysis controlling the water dissolved in the resulting fatty acid to a definite percentage, then without substantial reduction of temperature and pressure or exposure of the fatty acids to oxidation combining said fatty acids with alkali solutions in combining proportions and containing a definite percentage of water the total combined water being not more than will form a neat soap, said fatty acids and alkali being maintained in a state of flow, and finally discharging the resulting soap at a temperature between 250 to 400 degrees F. into a zone of lower temperature and pressure.

2. That process for continuously producing soap from fats suitable for soap making and alkaline reagents for the saponification thereof which consists in first causing the fat to flow counter-current to water in a suitable vertical chamber at a high temperature under sufficient pressure to maintain the water in a liquid condition, the period of contact of fat with water being sufficient to cause a substantial splitting of glycerin from the fatty acids, second continuously removing the fatty acids with the water dissolved in same and the sweet water resulting from said treatment in separate streams, third continuously commingling in an enclosed space under pressure the fatty acids without substantial reduction of temperature with an alkaline reagent and water, the said water thus added together with the water dissolved in the fatty acids being substantially no more than enough to result in the formation of neat soap, and the said alkaline substance being in substantially combining proportions, and fourth delivering the resulting soap at a temperature between 250 and 400 degrees F. to a point for cooling the same.

VICTOR MILLS.