Oct. 15, 1940. B. CLAYTON 2,218,279

APPARATUS FOR RECOVERING GLYCERIN

Original Filed Nov. 3, 1937 2 Sheets-Sheet 1

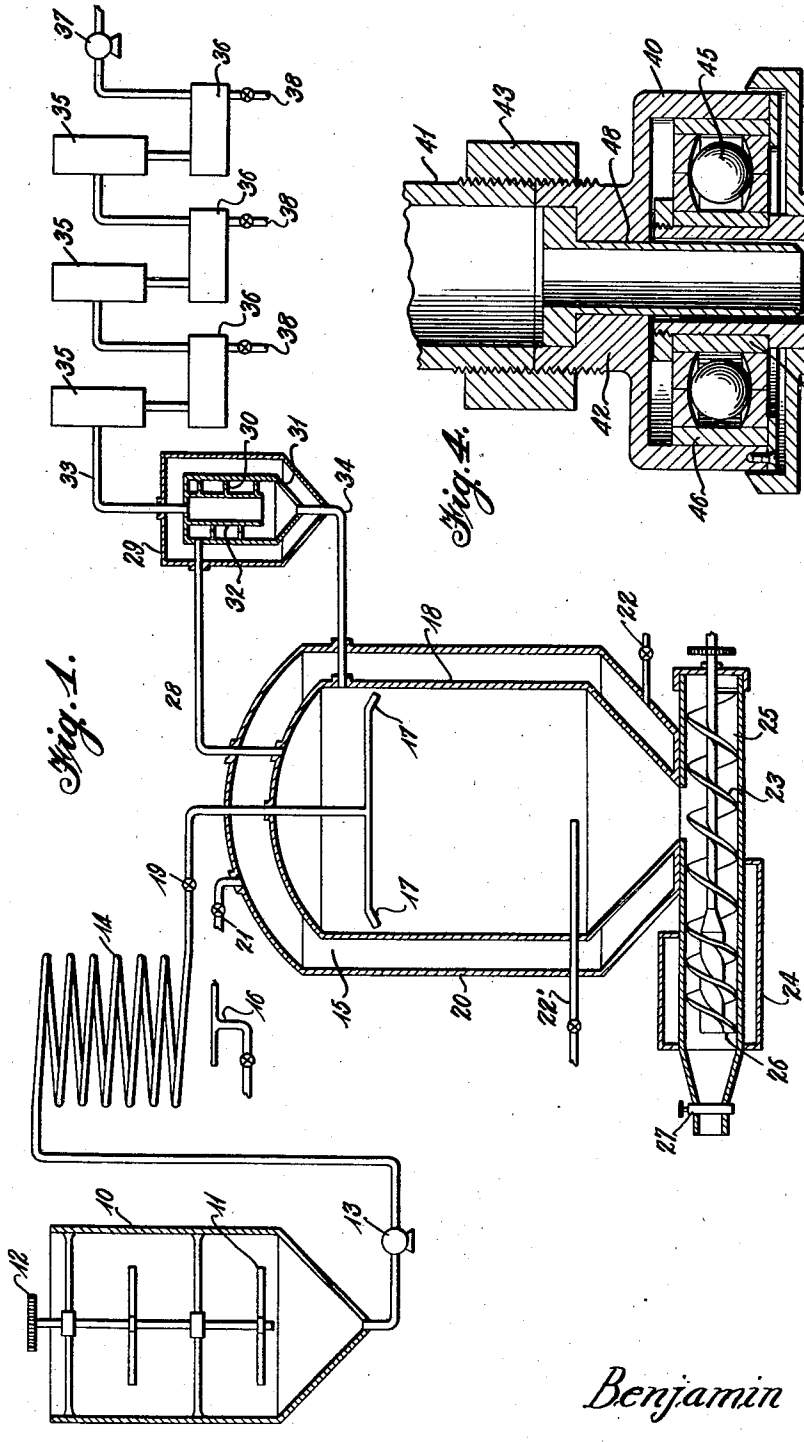


Fig. 1.

Fig. 4.

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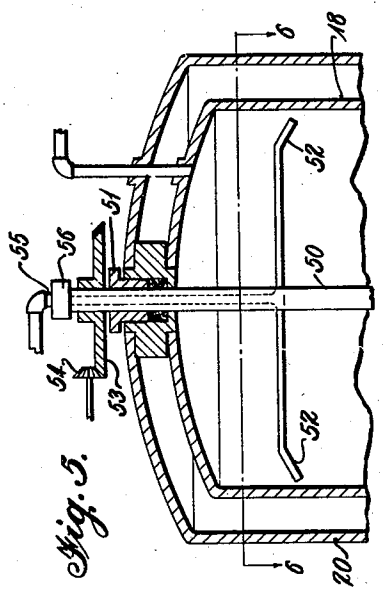


Fig. 5.

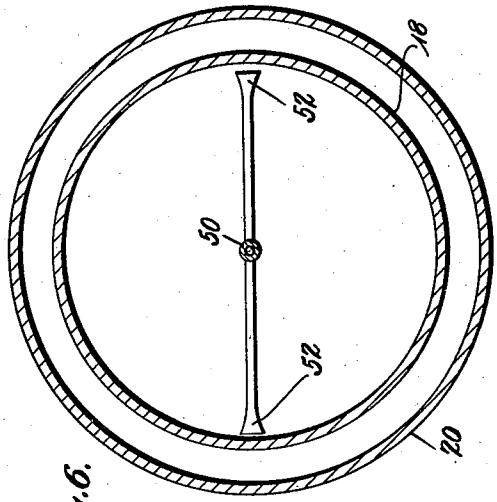


Fig. 6.

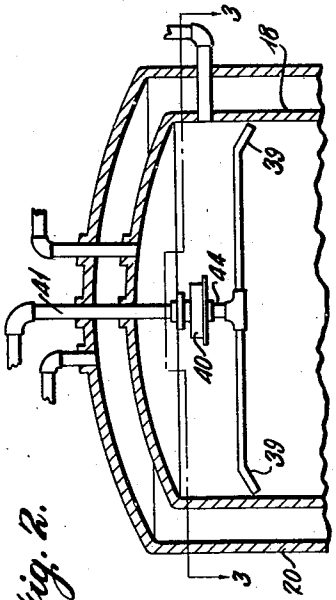


Fig. 2.

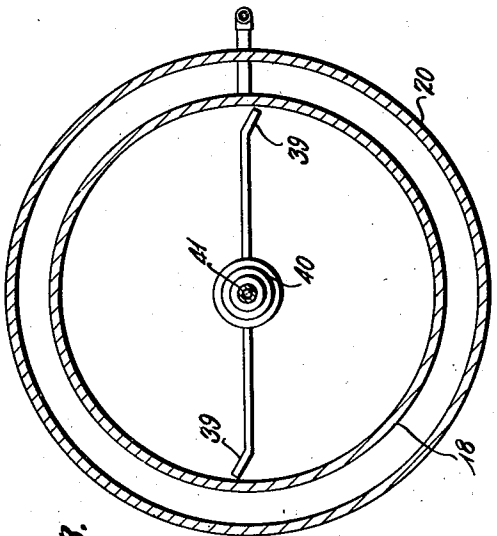


Fig. 3.

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Patented Oct. 15, 1940

2,218,279

UNITED STATES PATENT OFFICE

2,218,279

APPARATUS FOR RECOVERING GLYCERIN

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Original application November 3, 1937, Serial No. 172,644. Divided and this application April 11, 1938, Serial No. 201,443

1 Claim. (Cl. 252—371)

This invention relates to a process and apparatus for recovering glycerin and more particularly to a process and apparatus for recovering glycerin from admixture with soap.

In the production of soap from fats and oils containing glycerin in combination, relatively large proportions of glycerin are produced and this glycerin constitutes a valuable by-product. The conventional process of separating and recovering the glycerin from the soap mixtures is by adding salts such as sodium chloride to the mixture to cause the same to separate into layers, one consisting essentially of soap and the other including water, glycerin and added salt. The two layers are separated by decantation and by this means glycerin liquor containing large amounts of salt and water is produced. The glycerin liquors are then evaporated to remove a large part of the water and to cause part of the salt to crystallize out. The partially concentrated glycerin liquors are then concentrated by distillation to produce a high glycerin content. During this stage the presence of large amounts of salts which are poor heat conductors results both in large expenditures of heat and in superheating and requires the application of high temperatures for extended periods of time, all of which causes a decomposition of glycerin into products like acrolein, trimethylene glycol and other decomposition products.

In accordance with the present invention, the soap mixture is heated out of contact with the atmosphere to a temperature sufficiently high to cause the glycerin to be evaporated under vacuum conditions and the glycerin is thereby directly separated from the soap in relatively pure form without the addition of any salts or other materials for causing separation of the glycerin. The soap mixture treated in accordance with the present invention may be produced by the conventional batch processes but may be advantageously produced in a continuous process such as disclosed in the copending application Serial No. 119,168, filed January 5, 1937, now issued as Patent No. 2,190,615 of February 13, 1940. Such continuous processes of making soap mixtures involve the continuous mixing of oil or fat and alkali solutions at elevated temperatures and out of contact with the atmosphere to produce soap and glycerin. Such a soap mixture or one produced by the conventional batch process may be heated to a glycerin vaporizing temperature and delivered into a vacuum chamber wherein the glycerin is volatilized and removed in vapor form from the soap. The escape of glycerin from the soap is a function of temperature, time and amount of vacuum. It is generally necessary to heat the soap to a temperature at which it is liquid after the glycerin has been removed in order to effect substantial complete volatilization of the glycerin. Sufficient time must be provided for the glycerin to separate from the soap, but subjection of the soap and glycerin mixture to high temperatures for extended periods of time tends to decompose the glycerin and damage the soap.

It is, therefore, an object of the invention to provide a process of separating glycerin from soap under high temperature conditions in which the glycerin is rapidly separated from the soap before decomposition of the soap or glycerin is produced.

Another object of the invention is to produce substantially pure glycerin directly from mixtures containing soap and glycerin.

In order to cause rapid separation of glycerin from the soap, it has been found advantageous to spread the heated mixture in thin films upon surfaces in a vacuum chamber. Preferably these surfaces are heated so as to maintain the temperature of the heated mixture.

It is, therefore, another object of the invention to provide a process of separating glycerin from soap by spreading a heated soap and glycerin mixture in thin films upon surfaces in a vacuum zone.

Another object of the invention is to separate glycerin from heated soap and glycerin mixtures in a vacuum chamber by spreading the same in thin films upon heated surfaces.

This operation is preferably accomplished by directing the heated mixture against heated walls of a vacuum chamber by means of a nozzle or other opening. The particles as they strike the wall form a film of soap which slides down the walls. The film is an efficient means of exposing a large portion of the soap mixture from which the soap can be released. By controlling the distance through which the soap mixture travels down the walls of the chamber, sufficient time can be provided for substantially complete separation of glycerin without substantial damage to the soap or glycerin.

This method of evaporating glycerin from a soap mixture produces entirely different results than merely spraying the mixture of soap and glycerin into the chamber. While the film travels down the sides of the chamber only its clean surface is in contact with the kettle atmosphere, whereas the particles of soap formed by spraying have their clean surfaces exposed to the kettle atmosphere for only a comparatively short time before they fall to the bottom of the chamber. In spraying operations, mounds or heaps of soap build up on the bottom of the chamber, and this interferes with the proper volatilization of the glycerin from the soap. A much longer time must be provided to reach the desired degree of volatilization of the glycerin and the soap must be exposed to the chamber temperatures for

longer times. Also small soap particles formed by spraying are blown over or carried with the vapors and the soap in the bottom of the chamber does not release volatiles smoothly but sudden explosive upheavals occur in the soap mass resulting in the blowing up of soap particles which are entrained in the vapors removed from the chamber.

By directing the soap against the walls of the chamber a downward flowing film of soap is produced which loses volatiles as it travels down the sides of the chamber. There is no building up of soap particles or superposed soap films resulting in explosive liberation of vapors. The carrying over of soap particles with the vapors is thereby greatly minimized and the volatilization of glycerin takes place smoothly and rapidly.

A particularly effective way of discharging the mixture of glycerin soap against the wall of the vacuum chamber is to use revolving nozzles. The revolving nozzle makes efficient use of the kettle as the entire circumference of the surface of the walls is coated with a thin downwardly flowing film of soap from which the glycerin is rapidly and smoothly released. Stationary discharge nozzles which direct the mixture against the walls of the chamber are also contemplated by the present invention, although only that surface of the kettle within the fan-like spray is utilized in the volatiliaztion of glycerin from the soap.

It is, therefore, an object of the invention to provide a process and apparatus in which the entire circumference of the walls of a vacuum chamber is employed for glycerin volatilization.

Other advantages and objects of the invention will appear in the following description of preferred embodiments of invention described in connection with the attached drawings, of which:

Figure 1 is a schematic view of a complete apparatus for recovering substantially pure glycerin from soap;

Figure 2 is a fragmentary view in section of an evaporating chamber showing one type of revolving nozzle;

Figure 3 is a horizontal section taken on the line 3—3 of Figure 2;

Figure 4 is a fragmentary sectional view of the bearing arrangement of the nozzle of Figures 2 and 3;

Figure 5 is a view similar to Figure 2, showing a modified form of revolving nozzle; and

Figure 6 is a horizontal cross-section taken on the line 6—6 of Figure 5.

Referring more particularly to the drawings, in Figure 1, 10 indicates a source of supply of soap mixture which may be a conventional soap kettle provided with an agitator 11 driven from any suitable source of power through a pulley 12.

As indicated before, any other suitable source of soap mixture may be employed, for example, a continuous saponification process disclosed in application Serial No. 119,168 now issued as Patent No. 2,190,615 of February 13, 1940, referred to above. The soap mixture may be pumped by a high pressure pump 13 through a heating device shown as a coil 14 and into an evaporating chamber 15. The temperature of the mixture is raised in the heating coil 14 by any suitable means such as a burner 16 for gaseous or liquid fuel, to a temperature sufficiently high to cause glycerin to be evaporated in the evaporating chamber 15. While only one coil 14 is shown, two or more such coils in which the mixture is brought to successively higher temperatures in series may sometimes be desirable along with additional pumps between the coils to force the mixture therethrough. The temperature reached in coil or coils 14 will depend upon the type of soap being processed but will usually fall between temperatures of 450° and 620° F. These temperatures are usually above the decomposition point of the materials from which the soap was made, and care must be taken that substantially complete saponification is obtained before subjecting the mixture to such high temperatures. In Figure 1, stationary nozzles 17 are shown and the soap mixture is discharged against the wall 18 of the evaporating chamber 15. The soap mixture flows down the wall 18 in a thin film such that the glycerin vapors are rapidly and substantially completely liberated. Relatively high superatmospheric pressures are usually employed in the coil 14 to prevent too great vaporization therein. Thus pressures ranging from 50 to 1000 pounds imposed by the pump 13 and maintained by restricted orifices in the nozzles 17, by a valve 19 in the pipe leading from the coil 14 to the evaporating chamber 15 or by making the pipe of coil 14 sufficiently small, may be employed in the coil 14 depending upon the amount of vaporization desired therein. By employing pressures in the upper portion of the range above given, the mixture may be maintained substantially all in liquid phase in the coil 14, but in general it has been found preferable to provide at least partial vaporization of glycerin in the coil 14 in order to increase the amount of heat which may be imparted to the mixture therein.

As any vaporization of glycerin in the evaporating chamber 15 tends to cool the film of mixture upon the walls 18, it is generally necessary to supply additional heat to the mixture in the evaporating chamber 15. This can be conveniently done by enclosing the evaporating chamber within a heating jacket 20 through which any desired heating medium such as steam or heated mineral oil may be circulated by means of the pipes 21 and 22. Additional heat for vaporization can thereby be supplied directly to the film of mixture upon the wall 18 and the soap may be maintained in liquid form after water and glycerin vapors have been removed therefrom so that it will flow downwardly into the lower portion of the evaporating chamber 15. Also additional heat may be supplied by introducing steam, preferably superheated to a temperature at least as high as that of the mixture leaving the coil 14, into the vacuum chamber, for example, by the pipe 22'.

In order to promptly remove the soap from the evaporating chamber and to cool the same, a screw conveyor 23 provided with a cooling jacket 24 can be conveniently employed. Such a device is effective to continuously discharge the substantially anhydrous soap from the evaporating chamber without breaking the vacuum and also to cool the soap below a temperature at which it would be damaged by contact with the air before contacting the soap with the air.

The housing 25 of the conveyor 23 forms a part of the evaporating chamber 15 and the soap is delivered through a restricted passage formed by enlarging the end portion 26 of the conveyor so as to plug the conveyor housing against entrance of air. A valve 27 may be provided in the discharge end of the conveyor housing so that the vacuum may be maintained during starting and stopping of the apparatus when no soap is present in the conveyor housing 25.

The vapors are withdrawn from the evaporat-

ing chamber through a pipe 28 and may be delivered to an entrainment separator 29. It will be noted that discharging the mixture against the walls of the chamber 15 and causing the same to flow downwardly along the walls, provides a substantially unimpeded path for the vapors upwardly through the evaporating chamber 28. The vapors are thus not required to pass through a spray of material and entrainment is largely prevented. Entrainment separator 29 has been illustrated as including a helical baffle 30 formed between the casing 31 of the entrainment separator and an inner baffle 32 so that the vapors passing through the separator are forced to follow a helical path and then make an abrupt turn upwardly through the baffle 32 to the vapor withdrawal pipe 33. Thus entrained liquids or solids are thrown out of the vapors and are conducted back to the evaporating chamber through the pipe 34.

One or more condensers 35 provided with receivers 36 are provided for condensing the glycerin water vapors withdrawn from the vacuum chamber 15 either in a single condenser or in any desired fractions in several condensers. A vacuum is maintained in the receivers, condensers and evaporating chamber 15 by means of a vacuum pump 37 connected to the last receiver 36. It is desirable to maintain as high a vacuum as commercially practicable in the evaporating and condensing system. Thus, vacuums ranging from 26 to 29 inches of mercury have been successfully employed. Condensate may be withdrawn from the receivers 36 through pipes 38 by any suitable means, for example, pumps (not shown).

The structure shown in Figures 2, 3 and 4 provides rotating nozzles 39 by which the heated soap mixture may be distributed around the periphery of the wall 18 of the evaporating chamber. The soap mixture may be delivered into the chamber with considerable velocity and by radially inclining the nozzles 39 as shown in Figure 3 and pivotally connecting the nozzles at 40 with the pipe 41 delivering the mixture into the chamber, the nozzles can be caused to revolve. A suitable rotary connection for the nozzle is shown in Figure 5 and may include a stationary element 42 to which the pipe 41 is connected by means of a threaded member 43. A pipe 44 supporting nozzles 39 is rotatively secured to the member 42 by an anti-friction bearing including bearings 45, a bearing race 46 secured to the stationary member 42, and a bearing race 47 secured to the rotary pipe 44. A nipple 48 is secured to the stationary member 42 and extends downwardly within the rotary pipe 44 and a flange 49 may be secured to the rotary pipe 44 for preventing entrance of extraneous material into the bearing races 46 and 47. By this structure the nozzles 39 will continuously revolve in the same manner as a reaction turbine.

In Figures 5 and 6 is shown a device in which rotation of the nozzle is produced through external gearing so that the speed of the same may be accurately controlled. Thus, a rotating member 50 extending through a packing gland 51 in the wall 18 of the evaporating chamber may carry rotating nozzles 52. The shaft 50 and nozzles 52 may be rotated by a bevel gear 53 secured to the shaft 50 and driven by a pinion 54. By providing a hollow bore in the shaft 50 and connecting the pipe 55 which delivers the heated material to the evaporating chamber to the shaft 50 through any suitable rotary connection 56, the heated mixture may be delivered continuously around the periphery of the wall 18 of the evaporating chamber at any desired speed depending upon the speed of the pinion 54. If desired, the shaft 50 may continue downwardly and drive a scraper (not shown) in the lower portion of the evaporating chamber in order to insure that the soap is continuously delivered into the conveyor housing 25.

Examples of the products resulting from the present process are given in the following table:

Table I

Run	Amount of soap produced	Amount of total glycerin condensates	Percent of glycerin in condensates	Percent fatty matter, including soap in glycerin condensates
	Pounds	*Pounds*		
1	449	302.75	11.0	0.34
2	615	373.75	11.7	0.37
3	760	432.0	13.7	0.346
4	565	248.0	12.6	0.37
5	429	268.5	10.6	0.19
6	426	290.5	13.1	0.097

The amount of total glycerin condensates represents all of the condensate collected in the condenser system, and it will be noted that the amount of these total condensates is always much less than the amount of soap produced, in most cases being only slightly more than 50% of that amount. This is contrasted with the conventional method of salting out glycerin, in which the glycerin waters or spent lyes range from an amount approximately equal to the soap produced to three times as much or more. This means that much less water must be evaporated in the present process to produce a concentrated crude glycerin. Also the total impurities in the total glycerin condensates are much less than 1% since there is no salt present as contrasted with the 5 to 15% of salt and the 1% or more of non-volatile organic impurities present in the glycerin water from conventional glycerin separation steps. This is summarized in the following table:

Table II

	Combined condensates of present process	Spent lye liquors
Percent glycerine	11–18%	3–10%.
Amount of salt	None	5–15%.
Organic impurities	From less than 0.09%–0.40%.	More than 1%.

As an example of the composition of the various condensates which can be obtained from a condenser system employing four condensers, the following table relating to Run #1 of Table I is given:

Table III

No. of condenser	Pounds of condensate	Percent of glycerin in condensate	Percent fatty matter, including soap in condensate
1	5.25	81.8	2.48
2	24.0	24.5	0.81
3	257.0	9.0	0.28
4	16.5	1.0	0.0

It will be noted that a large percentage of the impurities was collected in the first condenser which was nearest the evaporating chamber and that the other condensates are relatively pure.

The bulk of the condensate was condensed in condenser #3 which contains 9% glycerin and only 0.28% of impurities.

When the glycerin water from the conventional glycerin salting out processes are chemically treated to remove impurities and are evaporated at the expense of large amounts of heat to concentrate the glycerin and crystallize out large amounts of salts, large amounts of impurities remain therein. The composition of three crude glycerins obtained from such processes are given by the following table:

Table IV

	I	II	III
	Percent	*Percent*	*Percent*
Percent glycerol	82.54	79.18	56.47
Non-volatile organic matter	1.16	3.45	14.55
Ash	8.28	9.68	19.66

A comparison of such crude glycerins from such conventional processes and the total condensate from the present process evaporated to approximately 80% glycerin is given in the following table:

Table V

	Crude glycerin of present process	Evaporated crude glycerin
		Percent
Amount of salt	None	5–15
Non-volatile organic matter.	0.5–5.0%	1.0–15
Ash	From less than 0.1–0.8%	8.0–20.0

In the above table, the amount of ash tabulated for the present process is from the non-volatile organic matter, chiefly soap, as there are no inorganic salts present, while the ash for conventional evaporated crude glycerin is from both the salt and non-volatile organic matter present. It is a comparatively simple matter to obtain substantially pure highly concentrated glycerin from the crude glycerin of the present process by distillation because of the high purity of the crude glycerin, but the amount of salt and organic impurities of the evaporated crude glycerin from salting out processes renders the distillation thereof extremely difficult. Large amounts of these impurities accumulate in the still, cause local overheating of the glycerin in the still, and result in thermal decomposition thereof to produce products such as acrolein, trimethylene glycol, etc., which products are present in the condensed glycerin and are difficult to remove therefrom.

Also, the present process provides for an almost complete separation of the glycerin from the soap. Soap containing much less than 1% glycerin has been consistently produced by the present process. The amount of glycerin retained in the soap, if a glycerin carrying soap is desired, may, however, be easily controlled by varying the temperature at which the soap mixture is introduced into the evaporating chamber, the temperature or vacuum maintained therein, or the length of time the soap remains in the evaporating chamber or any combination of these conditions.

This application is a division of my copending application Serial No. 172,644, filed November 3, 1937, now issued as Patent No. 2,195,377 of March 26, 1940, Process and apparatus for recovering glycerin.

While I have described the preferred embodiments of the invention, it is understood that the details thereof may be varied within the scope of the appended claim.

I claim:

A high speed apparatus for making soap and recovering relatively pure glycerin comprising, in combination: a heating device, means for discharging a saponified mixture of soap and glycerin from said heating device as a continuous stream, an evaporating chamber, a spraying nozzle adapted to direct said stream along the interior surface of the wall of the said evaporating chamber so that the same descends slowly as a film thereby permitting sufficient time to vaporize substantially all of the glycerin therefrom without decomposition to the soap, means for withdrawing said vaporized glycerin and condensing the same, means for introducing superheated steam into said evaporating chamber to assist in the removal of the glycerin vapors from said chamber and maintain the temperature of said soap above the melting point when anhydrous, said spraying nozzle being constructed and arranged so that the superheated steam and the vaporized glycerin ascend within said chamber without colliding with the saponified mixture being introduced thereto whereby the glycerin removed and cooled is substantially free of foreign matter.

BENJAMIN CLAYTON.