UNITED STATES PATENT OFFICE.

PETER KREBITZ, OF MUNICH, GERMANY.

MANUFACTURE OF SOAP.

No. 858,295. Specification of Letters Patent. Patented June 25, 1907.

Application filed November 2, 1904. Serial No. 231,086.

To all whom it may concern:

Be it known that I, PETER KREBITZ, a citizen of the Empire of Germany, and residing at Munich, in the Kingdom of Bavaria and the Empire of Germany, have invented certain new and useful Improvements in the Manufacture of Soap; and I do hereby declare the following to be a full, clear, and exact description of the invention, such as will enable others skilled in the art to which it appertains to make and use the same.

This invention relates to processes of making soap and consists in a method of making soap by an organized succession of steps, each separately useful, and as a whole cooperating to manufacture soap in a cheap and efficient manner; all as more fully hereinafter set forth and claimed.

In the present method an insoluble soap is first prepared in a manner permitting both the production of an article of great purity and desirable characteristics and the production of a glycerin solution, or "sweet water," of good strength and purity. The insoluble soap is then converted into the ordinary potash or soda soluble soap in a cheap, efficient and thorough manner.

The base of the insoluble soap may be lime, strontia, baryta or zinc oxid, but for the purpose of simplicity of description, lime will be more particularly referred to hereinafter.

In the ordinary methods of making insoluble soap, the fatty matter is usually heated with water and lime or other base and the heat maintained at or above the boiling point of water, stirring continuously, until saponification is completed. Aside from the heat and power required in this operation, the resulting product is of undesirable physical characteristics, being a hard, caked mass, the lime soap being usually known as "lime rock." In order to extract glycerin from it, in case no large excess of water has been used, it must be disintegrated, and disintegration is necessary in any event before it can be efficiently converted into soluble soap. Using an excess of water, the glycerin forms an undesirably dilute solution. In the first step of the present method, on the other hand, an insoluble soap is obtained which is very loose and porous in texture, needing no special disintegration and containing the glycerin as a comparatively concentrated, readily removed solution. By proper washing, a sweet water containing 12 to 30 per cent of glycerin may be directly obtained. Both because of the open texture of the soap and because the saponification is more far-reaching and complete than in the stated prior art, the glycerin is obtained in larger quantity. To produce this loose-textured lime or other soap, the fat is first heated to 100° C., or thereabout and with it is thoroughly incorporated the necessary amount of lime, or other base. In the case of lime, 10 to 14 per cent of the weight of the fat may be employed. With the lime should be employed a certain small amount of water, from half the bulk of fat to an equal quantity. This water may be very advantageously replaced by dilute glycerin solution, later obtained. The mixture of lime, fat and water is heated to the boiling point of water, to begin the reaction, and then in lieu of being continuously heated and stirred, as in the prior art, is merely allowed to stand, no further heat being applied. If the operation be performed in a heated kettle, heating is discontinued when the temperature reaches the necessary point and the cover of the kettle removed to permit free evaporation and consequent cooling. During the gradual cooling which ensues, the reaction initiated in the heating operation spreads throughout the mass, after a time becoming complete. With ordinary fats this time is from five to ten hours. By addition to the fat of free fatty acid or resin, the reaction can be much accelerated, being complete in from half an hour to an hour. Five per cent is a suitable quantity. The insoluble soap so produced is of very loose texture and is readily comminuted by grinding, crushing and so on. All the glycerin of the fat, together with that of the sweet water, if the latter was employed in saponification, is found in comparatively concentrated form in the wetting film on the surfaces of the granules composing said loose textured mass, and it may be readily removed by the use of small quantities of wash water. Hot water should be used and methodical washing resorted to, several portions of water being employed. The last wash, representing a weak glycerin solution, is preferably reserved for saponifying more fat in the manner described.

The lime soap after washing is free from all soluble impurities and is ready for conversion into soluble soap. This is preferably done by solutions of alkali carbonates.

Owing to the open texture of the lime soap so produced conversion proceeds more readily and thoroughly than with the usual lime rock, and owing to its purity a better grade of soap is produced. For a number of reasons, I prefer to use an excess of sodium or potassium carbonate over that theoretically necessary for the conversion of the lime soap. With the calculated amount of carbonate, owing to the laws of mass action, conversion is never complete and the reaction is very slow. And the precipitated carbonate of lime not only contains an admixture of lime soap but in some manner a portion of the soluble soap formed is also rendered insoluble and carried down; perhaps as a double salt. These losses aggregate very great amounts; sometimes as high as 10 to 30 per cent of the soap. Further, using the molecular amounts the lime carbonate is hard to wash free of soap; probably from the presence of the stated double salts. By the use of 2 to 6 per cent of carbonate over the theoretical amounts, the stated sources of loss are avoided and the added excess is readily regained in later operations. As facilitating conversion and separation from the calcium carbonate, some salt is preferably used with the sodium carbonate. The carbonate may be directly dissolved in 15 per cent salt lye; which may be one resulting from a previous similar operation. The water present in this operation should be two or three times the quantity of soda. From 5 to 20 per cent of salt may be present in the soda solution.

The alkali solution, or salt and soda solution, is brought to a boil and the lime soap thoroughly incorporated. Double decomposition takes place and calcium carbonate is precipitated, the speed of the reaction depending somewhat on the state of comminution of the insoluble soap. During its progress, the said insoluble soap should be evenly distributed throughout the mass and the temperature should be kept at the boiling point. Care should be taken that the mass does not become too thick, and retains a certain mobility. Thickening may be prevented by suitable additions of salt or strong salt solution.

After the conversion is complete, in the case of soda soap, the soluble soap is salted out by a sufficient addition of salt. The curd soap floats and the carbonate of lime separates below the salt lye.

Salt lye and lime carbonate are separated in any suitable manner, as, for instance, by the use of a filter press or a centrifugal machine. The carbonate is then washed with several portions of hot water. As it carries down with it a certain amount of the excess of soda carbonate used, extraction of the adhering soap is easy and complete without hydrolysis. In second and third wash waters, addition of a little salt is desirable to prevent hydrolysis. About 2 to 3 per cent of salt in these wash waters is a desirable quantity. These wash waters may be used to purify the curd soap, or they may be employed in dissolving carbonate for decomposing the lime soap. Or, if desired, the fatty acids of the soap in the second and third washings may be thrown out by the addition of a little acid, such as sulfuric. The salt lye separated from the calcium carbonate may have its excess of alkaline carbonate converted into soap and separated by treatment with a little fatty acid or resin, or it may be used, after addition of water and more carbonate of soda, for the decomposition of more lime soap. The same salt lye may be used for a number of boils in this manner, since the separation of the calcium carbonate serves to carry down much coloring matter and other impurities and thereby purify it.

In proceeding according to the foregoing method, the separated calcium carbonate should not contain more than 0.4 to 0.6 per cent of soap, and the soda soap obtained should not contain more than, at the highest, 0.02 per cent of calcium carbonate.

In the manufacture of potash soap, the conversion should be carried on with carbonate solution alone, as salt would form soda soap, and the solution of potash soap is separated from the calcium carbonate by means of a filter press, or other suitable means. Excess of carbonate in the solution is neutralized by the addition of fatty acid or resin in the described manner.

Soaps made by the described procedure are particularly light in color and possess detergent qualities as good as any other soap from the same fats; in many cases, being even superior in adhesiveness and scouring qualities. Their durability is good; they bleach rapidly and they do not become rancid and dark, even after considerable periods of time. The glycerin is obtained in a concentrated state, of good purity and in large quantity. From the physical properties of the lime soap, its conversion into soluble soap is rapid and complete, using the described method. Omission of the usual prolonged heating and stirring economizes in labor, and fuel and obviates the necessity of much costly machinery. The loose textured character of the lime soap, also contributes to the saving in power and machinery. This loose porous texture of the insoluble soap not only permits perfect extraction of the glycerin in a concentrated state but also allows at the same time a very thorough elimination of soluble impurities, so that the washed soap is very suitable for making high grade soluble soap. The absence of these impurities contributes materially to the success of the subsequent operations as does also the open texture.

Having thus fully described my invention,

what I claim as new and desire to secure by Letters-Patent of the United States is:—

1. The process of making an insoluble soap which consists in thoroughly incorporating hot fat and a suitable base forming insoluble soaps with a limited quantity of water, heating the mixture to start the reaction and in then allowing the mixture to stand and cool while said reaction progresses.

2. The process of making a lime soap which consists in thoroughly incorporating hot fat with lime and a limited quantity of water, heating the mixture to start the reaction and in then allowing the mixture to stand and cool while said reaction progresses.

3. The process of making an insoluble soap which consists in thoroughly incorporating hot fat and a suitable base forming insoluble soaps with a limited quantity of water containing glycerin, heating the mixture to start the reaction, and in then allowing the mixture to stand and cool while said reaction progresses.

4. The process of making lime soap which consists in thoroughly incorporating hot fat with lime and a limited quantity of water containing glycerin, heating the mixture to start the reaction and in then allowing the mixture to stand and cool while said reaction progresses.

5. The process of making an insoluble soap which consists in thoroughly incorporating hot fat containing a free acid with a suitable base forming insoluble soaps and a limited amount of water, heating the mixture to start the reaction, and in then allowing the mixture to stand and cool while said reaction progresses.

6. The process of making lime soap which consists in thoroughly incorporating hot fat containing free acid with lime and a limited amount of water, heating the mixture to start the reaction and in then allowing the mixture to stand and cool while said reaction progresses.

7. The process of making an insoluble soap which consists in thoroughly incorporating hot fat containing a free acid with a suitable base forming insoluble soaps and a limited amount of water containing glycerin, heating the mixture to start the reaction, and in then allowing the mixture to stand and cool while said reaction progresses.

8. The process of forming lime soap which consists in thoroughly incorporating hot fat containing a free acid with lime and a limited amount of water containing glycerin, heating the mixture to start the reaction, and in then allowing the mixture to stand and cool while said reaction progresses.

9. The process of making soap which consists in thoroughly incorporating hot fat with a base forming an insoluble soap and with a limited quantity of water, heating the mixture to start the reaction, allowing the mixture to stand and cool while the reaction progresses, washing it free of glycerin and soluble impurities and decomposing by boiling with a lye containing an alkaline carbonate in amount at least 2 per cent in excess of that equivalent to said insoluble soap.

10. The process of making soap which consists in forming a granular insoluble soap of a suitable base and decomposing said soap by boiling with a lye containing an alkaline carbonate in amount between 2 and 6 per cent in excess of that equivalent to said insoluble soap.

11. The process of making soap which consists in forming a granular insoluble soap of a suitable base and decomposing said soap by boiling with a lye containing sodium carbonate in amount between 2 and 6 per cent in excess of that equivalent to said insoluble soap and also containing a few per cent of salt.

12. The process of making soap which consists in forming an insoluble soap of a suitable base and decomposing said soap by boiling with sodium carbonate dissolved in a salt lye resulting from a previous operation of the same character, said sodium carbonate being in amount at least 2 per cent in excess of that equivalent to said insoluble soap.

13. The process of making and recovering soap which consists in forming an insoluble soap of a suitable base, decomposing said soap by boiling with a lye containing an alkaline carbonate in amount between 2 and 6 per cent in excess of that equivalent to said insoluble soap, separating the insoluble carbonate formed and washing the same with hot water to remove and recover co-precipitated alkaline carbonate and soap.

14. The process of making soap which consists in forming an insoluble soap of a suitable base, decomposing said soap by boiling with a lye containing sodium carbonate in amount between 2 and 6 per cent in excess of that equivalent to said insoluble soap, adding salt in salting-out amount after completion of the decomposition, and separating the curd soap and insoluble carbonate formed.

15. The process of making soap which consists in forming lime soap, decomposing said lime soap by boiling with sodium carbonate dissolved in diluted salt lye resulting from a previous similar operation, said carbonate being in amount at least 2 per cent in excess of that equivalent to said lime soap, adding salt in salting out amount after completion of the decomposition and separating the curd soap and insoluble calcium carbonate formed.

16. The process of making soap which consists in forming lime soap by thoroughly incorporating hot fat, lime and a limited quantity of water, heating to start the reaction and allowing to stand and cool while said re-

action progresses, washing the so-formed lime soap free of glycerin and soluble impurities with hot water, decomposing said purified lime soap by boiling with a salt lye containing sodium carbonate in amount at least 2 per cent in excess of that equivalent to said lime soap, adding salt in salting out proportions after completion of the decomposition, and separating the curd soap and insoluble calcium carbonate formed.

In testimony whereof I hereunto affix my signature in the presence of two witnesses.

PETER KREBITZ.

Witnesses:

ULYSSES J. BYWATER,

ABRAHAM SCHLESINGER.