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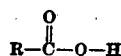
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PROCESS OF MAKING SOAP AND GLYCERINE

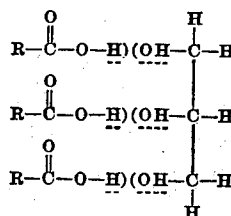
No Drawing.

Application filed February 19, 1931. Serial No. 517,098.

The invention relates to a new and useful process for the manufacture of soaps and glycerine represented by the general structural formula:



A glyceride molecule may be split so that with the proper distribution of the hydrogen and oxygen atoms of three added molecules of water there will result three molecules of fatty acid and one molecule of glycerine as represented structurally below.



This reaction actually takes place slowly when fats are boiled with water, resulting in the formation of fatty acids and glycerine. When pure water is used the reaction is, however, too slow to be economically advantageous. The progress of the reaction may be speeded up by adding to the water a small amount of mineral acid, as sulphuric acid, or a small amount of base, as lime, and by increasing the temperature of the reaction by carrying it out under pressure, as in the old autoclave processes. The process of aqueous saponification has also been hastened at ordinary pressures at the boiling point of water by the employment of special acid catalyzers, as in the well-known Twitchell process. In the autoclave processes it is impossible to employ very high temperatures to speed up the reaction without employing excessive and dangerous pressures and expensive apparatus. All of these processes have the disadvantage of depending on balanced reactions which do not run to an end and do not effect complete splitting of the fat into fatty acid and glycerine.

Fatty acids will combine readily with alkalies or the alkali of alkali carbonates to form soap in a manner akin to the usual interaction of acids and alkalies, or acids and alkali carbonates. Caustic alkalies act on

In the saponification of soap stocks two valuable co-products, soap and glycerine, are obtained. Of these two co-products soap is the more important and for this reason the methods of saponification known prior to my invention have been designed primarily to yield good soap. All of the known processes involve an appreciable sacrifice in both the quality and quantity of glycerine produced. The importance of glycerine as a product has grown so that for some years past this sacrifice in the yield of glycerine has constituted an appreciable but apparently unavoidable waste.

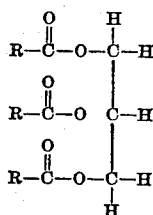
The raw materials from which soaps and glycerine are prepared are:

1. Fats and/or fatty oils of animal and/or vegetable origin.

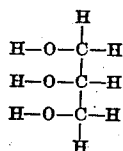
2. Basic or alkaline materials.

3. Additions of all sorts to the finished soap.

Fats and/or fatty oils contain substances known as glycerides. These are chemical combinations formed by the union of one molecule of glycerine with one or more molecules of fatty acid. A general structural formula for a typical glyceride is:

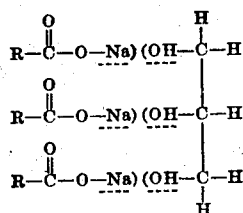


The structural formula for glycerine is:



Fats and/or fatty oils also contain varying proportions of free fatty acids which can be

fats and glycerides with the formation of soap and glycerine thus:



This reaction, known as saponification, is favored by heat, intimate contact and alkali concentration.

At temperatures up to the boiling point of water alkali carbonates exert only a slow saponifying action on fats and glycerides.

Fats and/or fatty oils generally contain small quantities of various saponifiable or unsaponifiable substances other than fatty acids and glycerides, which are very objectionable for soap-making purposes. These materials often cause extremely objectionable odors in the finished soap and exert an objectionable catalytic influence favoring the development of rancidity. Some of them also inhibit the lathering properties of the finished soap.

The most common method of soap-making now in use involves the saponification of comparatively high grade fats and oils in open or loosely covered soap pans or kettles with the aid of moderate steam heat and steam agitation at atmospheric pressure. During the process of saponification glycerine is formed. The mixture obtained by the saponification referred to above comprises soap, water, glycerine, excess alkali and usually some unsaponified fat. Glycerine is soluble in water and to a very considerable extent in soap, especially in soap containing an appreciable amount of water. For the removal of soap and excess alkali and other impurities the mixture of soap, glycerine, etc., obtained by saponification is salted out by addition to the soap kettle of considerable quantities of common salt or strong solutions of common salt. Strong salt solutions have the property of throwing soap out of solution while dissolving glycerine and water-soluble impurities. The soap, glycerine, salt, water mixture is boiled for some time and then allowed to settle. The glycerine, salt, water mixture containing some excess alkali, known as "soap lye", separates and is saved for recovery of glycerine. The glycerine divides up between the soap lye and the soap, and although it is somewhat more soluble in the soap lye than in the soap, a very considerable proportion is, nevertheless, retained by the soap phase, which is usually much larger than the lye phase, so that a considerable number of successive lye treatments or "washings" are necessary for the removal of the greater portion of the glycerine. Dur-

ing the latter part of the process the proportion of glycerine in the soap lye is very much reduced so that further washes are uneconomical, while an appreciable amount of glycerine is still left in the soap.

In order to recover glycerine from the soap lyes certain chemical treatments are necessary for the removal of excess alkali, soluble soaps and other impurities. It is also necessary to filter the lyes for removal of suspended impurities and to evaporate them for the removal of the large amount of water that is necessarily used in the "washes" for the extraction of glycerine. During the evaporation salt separates out before the glycerine becomes very concentrated and constitutes a troublesome impurity in the glycerine. Also, an appreciable loss of glycerine usually takes place in evaporation, due to overhead entrainment. The crude glycerine finally obtained by evaporation of the large amount of water used in extracting the glycerine is filtered or otherwise separated from the separated salt and is subjected to a series of distillations. The presence of salt in the crude glycerine and the accumulation of it in the still residue during the distillation of glycerine interfere to a considerable degree with the progress of the distillation and have an unfavorable effect on the quality of the distillate so that additional distillations are usually resorted to for further purification of the glycerine.

In every step of the soap and glycerine-making process losses of glycerine are encountered. A very appreciable amount of glycerine remains unextracted in the soap without adding to the good qualities thereof, and further quantities are lost during the filtration of the lye, the removal of the salt, and in the still residues, while other portions are unavoidably lost by entrainment in evaporation and in further distillations.

The soap, after having been subjected to a number of boilings with excess alkali to insure complete saponification, and a number of washes for the further extraction of glycerine, is still contaminated with an appreciable excess of alkali and a considerable amount of salt and contains an appreciable amount of glycerine that is not recoverable. The soap is boiled with water and brought to a condition of fluidity that favors the separation of the soap mass into two layers, the upper of which is known as "settled" soap and contains about three-fourths to four-fifths of the soap, and the other as "nigre", which contains most of the salt and excess alkali still left in the soap pan and the greater portion of insoluble impurities. The disposal of the nigre by reprocessing is an additional burden in known soap-making processes.

Another process somewhat in use is to subject the fats and/or fatty oils to the so-called

Twitchell process, which effects an acid saponification or hydrolysis of the fatty materials yielding fatty acids and glycerine, the glycerine formed in the reaction remaining dissolved in an aqueous acid layer and the fatty acids forming a layer by themselves. This process does not effect complete hydrolysis in a single treatment but gives a balanced reaction in which the final products comprise fatty acids and glycerine formed by the hydrolysis and an appreciable amount of unhydrolyzed fat or fatty oil. In order to carry the hydrolysis or acid saponification more nearly to completion, further treatments are necessary, all involving time, expense and chemical treatment of the aqueous acid layer containing glycerine, and the subsequent evaporation thereof and distillation of the glycerine therefrom to render it fit for use. This process also leaves an appreciable amount of unrecovered glycerine unless the fats are subjected to several prolonged treatments, in which case a sensible darkening of the product occurs due to atmospheric oxidation and other causes. In common practice there is also an appreciable loss of glycerine in operating by this process. The fatty acids formed in the process are then commonly subjected to soap-making treatments substantially similar to those outlined above.

In any of the common soap-making processes, such as I have described above, the bulk of water involved in the extraction of the glycerine is many times the bulk of the glycerine produced. The cost of handling and evaporating this extremely large bulk of water is high. The process of my invention may be so operated that the glycerine is produced in concentrated form without any intermediate steps in which large quantities of water must be handled and evaporated, and substantially all the glycerine produced may be so recovered. This decided simplification in the recovery of glycerine is a novel and decidedly advantageous feature of my invention.

I have discovered that soaps made from fats and/or fatty oils when properly heated and kept from contact with oxidizing agents can be raised to temperatures ranging from under 300 degrees C. to 350 degrees C., and even higher without pyrolysis or decomposition taking place. Saponification of the fats and/or fatty oils subjected to the above process may take place prior to this process or during it. I have also discovered that glycerine resulting from the saponification of fats and/or fatty oils under the above named temperatures and proper conditions of agitation can be separated substantially completely as a vapor from the saponification mixture. I have also discovered that aqueous hydrolysis brought about by steam agitation at the comparatively high temperatures mentioned is quite rapid in the presence of

basic media and that even when there is an insufficient amount of basic material present to convert all the fat and/or fatty oil into soap the hydrolysis will proceed comparatively rapidly with the formation of fatty acid and glycerine. I have further discovered in cases where I have employed a sufficient amount of basic materials to convert all fatty matter into soap that objectionable impurities often found in fats and/or fatty oils may be entirely removed from soaps made under the conditions of my invention. All of the above things I believe were unknown and not in use prior to my invention.

In carrying out my process I may employ a fat and/or fatty oil, as, for instance, tallow. This is introduced into a closed vessel constructed preferably in the shape of a still and provided with means for heating. I heat the fat to its melting point and begin to blow through it a strong current of steam, which serves the purposes of effectively agitating the contents of the still, driving air from the still, and guarding against the further entrance of any air. I prefer to heat the fat to a temperature well above the boiling point of water before introducing the alkaline or basic medium for saponification. I can employ a low temperature at the early stages of my process and subsequently increase the temperature, but prefer to introduce the alkaline or basic medium for fat saponification after first raising the temperature somewhat in excess of 200 degrees C., preferably 250 degrees to 300 degrees C. At these temperatures saponification takes place with extreme rapidity and the soap shows very little tendency to frothing or foaming, which may be observed and which may give some trouble at temperatures more nearly approximating the boiling point of water.

In carrying out my process I may employ alkaline media or bases either in solution or in substantially anhydrous form or mixed with insufficient water for complete solution. These may be added separately or mixed with the fatty material. Water or hydrated bases are usually considered necessary to saponification for the formation of fatty acids and glycerine or soap and glycerine. Ordinarily, water in aqueous condition is employed for saponification. Under the conditions that maintain in my process a quantity of liquid water is not necessary as the saponification is brought about with ease by water in vapor form, as steam. Where alkaline media or bases in solution are employed the water of solution evaporates with great rapidity, leaving little or no water except that in the form of steam. Where carbonates are employed carbon dioxide is evolved and the gas set free has an inert or protective action on the soap and/or fatty material, similar to steam.

The use of a strong current of steam to maintain very thorough mixing and to effectively prevent local overheating of any portion of the mass, and the maintenance of the temperature mentioned, comprise conditions very favorable to rapid saponification and to the volatilization of glycerine formed in the saponification. Under these conditions the soap mass or the mixture of soap and fatty material have a very low vapor pressure and glycerine has a much higher vapor pressure. Glycerine vapors are swept from the still by the strong current of steam almost as fast as formed. I prefer to continue the heating and agitation of the soap or the mixture of fat and soap until a temperature of about 300 degrees to 325 degrees C. is reached, at all times using a sufficiently strong current of steam to maintain thorough agitation and effectively exclude air. I prefer to use an alkaline or basic soap-making material sufficient to effect complete saponification or a slight excess over the amount necessary for complete saponification.

In the reaction carried out under these conditions glycerine, one of the products of the chemical reaction, is rapidly volatilized and, according to the well known chemical principle of mass action, the removal of glycerine in this way causes the saponification to run completely to an end, and the volatility of glycerine under the conditions stated permits of its complete removal from the reaction. In this reaction I may use caustic or carbonated alkalies, caustic or carbonated alkaline earths, nitrogen bases suitable for making soaps, or such other basic compounds of metals as may be necessary to produce metallic soaps where it is desirable to make this class of soap.

At a temperature approximating 300 degrees C. the soap mass is in a thin liquid condition and is perfectly stable where air is effectively excluded and local over-heating is prevented by the maintenance of sufficient agitation. The deleterious action of air and local over-heating may both be prevented by blowing a strong current of steam through the mass and carrying out the process in a still-like apparatus.

I may carry out my process at atmospheric pressure or at diminished pressures and as a matter of experience find certain advantages in operating at diminished pressures, particularly for the volatilization of the last traces of glycerine from the liquid mass of melted soap. In any event, I may readily realize high temperature without the use of high pressures and so I prefer to operate when using non-volatile bases without resorting to super-atmospheric pressures. I condense the glycerine vapors and may condense much of the water vapor that passes over with the glycerine. These may be condensed together or almost completely separated from

one another by fractional condensations, owing to the great difference in boiling points of water and glycerine and great difference in vapor pressures of these materials at any of the different temperatures used. Glycerine may be condensed in any concentration up to or even exceeding 99 per cent. while permitting water vapor to remain in the vapor phase. The portion of my process consisting of fractional condensation of glycerine from water vapor is a part of common practice in glycerine distillation and need not be described in detail.

My process may be carried out as a batch process, treating a given amount of fat with approximately the definite amount of alkali or basic material necessary to convert it into soap, or it may be carried out as a semi-continuous process by treating a certain amount of fat with the corresponding amount of alkali, distilling the glycerine therefrom, removing a portion of the soap thus formed, preferably while still in a hot, liquid condition, and adding further quantities of fats and alkali in such a way as to maintain the mass at the preferably reacting temperature at all times. I have specified the temperature of 300 degrees to 325 degrees C. as preferable temperatures. At this temperature the soap is stable, is in thin, liquid condition, glycerine in very volatile, the saponification takes place almost instantly, while agitation and thorough mixing without local over-heating are easily carried out. I am not limited to this range of temperature, however, as when thorough mixing and thorough exclusion of air are maintained, a temperature of 350 degrees C. may be employed or even exceeded for the reaction. On the other hand, the reaction described may be carried out at temperatures considerably under 300 degrees C., though it is preferable that the temperature be sufficiently high to maintain the substantially anhydrous soap in thin, liquid condition.

In my process I may utilize any of the fats or fatty oils commonly employed for soap-making purposes, but in addition to these or in place of them may use any of a great variety of fats or fatty oils which are poorly adapted for soap-making purposes by known methods because of one or another objectionable quality imparted by such fats or oils to soap made therefrom. I can, for instance, make good, firm soaps with good washing qualities, free from objectionable odor or color and with good keeping qualities, from such material as cod oil, herring oil, menhaden oil and whale oil, even when such oils are in a crude state and ordinarily yield soaps with repulsive odors, of dark color, of poor consistency and with poor washing properties, and with a strong tendency to deteriorate rapidly with age.

I am aware of the fact that certain objectionable fish oils and whale oil are at times

subjected to hydrogenation in attempts to make them more suitable for soap-making purposes, but the process of hydrogenation is relatively expensive and is not readily applicable to low grade oils or materials with a high content of free fatty acid, and soaps made from such hydrogenated fats possess extremely poor lathering and poor washing qualities if the hydrogenation is carried far enough to free the oils from objectionable odors and other qualities they naturally possess.

Furthermore, all attempts heretofore to make good soap-making materials out of such low grade fish oils and the like as do not yield readily to hydrogenation, which have consisted in hydrolyzing them and subjecting the fatty acids therefrom to purification by distillation, have been unsuccessful for the reason that such acids yield soaps improved in color only by otherwise possessing all the repulsive odor and other objectionable qualities given to soaps made from such oils by direct saponification.

My process does not consist alone in the improved saponification and recovery of glycerine detailed above, but effects other desirable results. One of these is the removal of certain undesirable unsaponifiable impurities by volatilizing them and removing them from the soap mass under conditions of the reaction. These impurities are to a certain degree common to all fats and fatty oils. They are present in very minor quantities and in less objectionable form in the better grades of animal and vegetable oils, such as prime tallow and high grade cocoanut oil. They are present in greater quantities and in more objectionable form in various types of greases, such as house grease, extraction grease and garbage grease, and they are present in even greater quantities and in very objectionable form in most types of marine animal oils, imparting to these oils objectionable fishy odors, which become more pronounced after saponification of such oils, and which become even more pronounced on the aging of soaps made from such oils. The conditions which I have described above that maintain in my process, and which are favorable to the volatilization of glycerine, are also effective in removing the above mentioned unsaponifiable impurities from soaps made from these oils by my process, thus resulting in the production of a soap with a neutral or mild, agreeable odor. In the case of fish oils which are treated by my process, I succeed not only in removing objectionable volatile impurities possessed of a bad odor, but effect at the same time a rearrangement in the molecules of the soaps formed from some of the more unsaturated acids present in fish oils, so that the resulting soaps have properties more desirable than the soaps obtained from such oils by direct saponification, in that they are possessed of a better odor and firmer texture and frequently possess better lathering qualities. This desirable effect is produced to a large degree on other soap-making materials treated by my process.

No sensible loss is brought about in subjecting fats and/or fatty acids to my treatment, the actual amount of acids or soaps remaining being substantially equivalent to the amount of fats and/or fatty acids or soap started with, there being no sensible decomposition or molecular breakdown or loss, such as is experienced in the Varrentrapp reaction or modifications of it, and there being no sensible polymerization to ether acids or bodies with a substantially higher molecular weight than those started with. The fatty acids obtained from the soap produced by my process commonly have a very slightly higher melting point than those obtained by direct saponification and acidification of the same material. This difference in melting point is seldom more than a fraction of a degree and is brought about by the rearrangement of molecules of some of the unsaturated acids present. The iodine values of acids obtained from fatty material subjected to my treatment are usually somewhat less than the iodine value of acids obtained by direct saponification of the same fats or oils. This is also due to a rearrangement of the double bonds in the molecules of some of the unsaturated acids. Cases are known to the science in which a compound having the unsaturated bonds in one position, has a substantially higher iodine absorption than an isomeric body of the same molecular weight with the same amount of unsaturation having the unsaturated bonds in a different position. Fatty acids having unsaturated bonds so situated in a molecule as to be capable of the ready addition of large amounts of iodine are well known to have a strong tendency to absorb oxygen, and soaps made from such acids have a stronger tendency to become rancid than soaps made from fatty acids that absorb lower amounts of iodine. The fact is that soap made by my process shows less tendency to become rancid or otherwise deteriorate than soaps made from the same material by old soap-making processes, and this fact is probably due to the rearrangement of double bonds in some of the molecules of unsaturated acid that is brought about by my process, and also due to the removal of certain unsaponifiable, easily oxidized materials from the soaps, and the removal of impurities which may act catalytically to further oxidation and the formation of rancidity.

In cases where fats and/or fatty oils are subjected to my treatment with an insufficient amount of alkali to effect complete saponification, the soap formed by the alkali which is used acts catalytically in the presence of the strong current of steam and high tempera-

tures preferably employed, to effect comparatively rapid hydrolysis with the formation of glycerine and free fatty acids, both of which distil over and condense. The glycerine and distilled fatty acids under these circumstances condense substantially together but may be separated from one another with great facility as they are almost mutually insoluble. The distilled acids thus formed are capable of being made directly into soap. Ordinarily, I prefer to use a slight excess of alkaline or basic medium over that necessary to effect complete saponification but do not limit myself entirely to this amount. In some cases, even where a slight excess of alkali is used over the amount required for saponification, failure to employ proper sequence or proportions in adding successive quantities of the reacting substances may result in distillation of some fatty acids from the reacting mass. Such a practice constitutes no more than a diversion from the preferred manner of operating without departing from the general practice employed. In some cases when operating on very impure, low grade fats, I may prefer to subject the fat or oil first to saponification and cleansing treatments by ordinary soap-making processes for the removal of certain impurities that may easily be removed in this way and which at the higher temperature of my reaction may break down and cause a darkening of the reacting mass. My process very commonly appreciably improves the color of the soap obtained by it over the color obtained from the same material by ordinary soap-making processes. In cases where fats or fatty oils are given a preliminary saponification by old, well-known methods for the removal of such impurities as may be removed by present processes, the portion of my process consisting of heating soap under described conditions and blowing steam through it effects sensible improvements in the quality of soap so treated in odor, consistency, washing qualities and keeping qualities and in color where readily decomposable impurities have been removed by such preliminary treatments. In the case of some fats and oils such readily decomposable impurities may be removed by a simple preliminary washing and settling with water or other simple known process for accomplishing a similar result. During the part of my process in which I remove unsaponifiable, objectionable matter by volatilization at high temperatures in a strong current of steam and preferably under diminished pressure, such unsaponifiable matter may be condensed with the glycerine in which it is substantially insoluble, and removed with ease from the glycerine. Although I prefer to collect the glycerine which distils over during the carrying out of my process in a state of high concentration by

resorting to fractional condensation, I may employ the type of condensation which will result in condensing glycerine and a considerable portion of the steam employed so as to give a more dilute glycerine solution. This may be separated with ease from unsaponifiable matter or fatty acids, both of which are substantially insoluble in glycerine or in dilute glycerine solution, and concentrated by simple well-known means to a concentrated glycerine. Glycerine as obtained by my process is of a sufficient degree of purity to be directly applicable to many uses. It may, however, be readily purified to a very high degree of purity by subjecting it to an additional distillation, as by the well-known process of vacuum distillation with the aid of steam under diminished pressure.

In removing all or a portion of the soap formed by my process after removal of glycerine and unsaponifiable matter, I prefer to draw it off in liquid state while taking suitable precautions to exclude contact of air with the hot soap. One way of doing this is to draw the liquid soap off through a valve and pipe, and steam may be used to free this pipe from air and keep it free. The soap may be discharged into and beneath the surface of a mass of water or hydrated melted soap and the amount of water employed need not be greater than that necessary to leave an amount of water in the finished soap substantially equivalent to that obtained in other soap-making processes. I may also use other means of drawing off the hot soap formed in my process in such a manner as to exclude air from it while it is at a temperature that would cause the air to exert a deleterious action, but these need not be detailed as they do not form a part of my present process. Soap obtained in this way is suitable for use by itself or may be made into toilet or laundry soap, or soap powders, by any of the well-known methods of utilizing soap.

There are some low grade fats and fatty oils which contain so much objectionable coloring matter that they are not fit to make directly into soap by any process without resorting to distillation of their fatty acids. My process effects sufficient improvement in certain low grade fatty matter so that distillation is rendered unnecessary, but it may be employed in exceptional cases in conjunction with distillation of fatty acids so as to effect improvements that may not otherwise be effected. For this purpose distilled fatty acids obtained by known methods may be subjected to my process, or, where desirable, fatty acids obtained from material treated by my process may be subjected to distillation.

I have also discovered that my process is applicable to other esters of fatty acids which on saponification yield soaps and alcohols other than glycerine. These esters, sometimes called waxes, are akin in several respects to

fats and fatty oils. Some of them, as for instance wool grease, are saponified incompletely or not at all under ordinary processes of soap-making but may be saponified readily and completely by my process with the elimination of the alcohols and volatile impurities, and such alcohols if valuable may be collected and utilized.

In using the term "fatty acids" hereinafter in the claims I refer not only to the true fatty acids but also to similar acids which are unsaturated in varying degrees but which may also be separated from natural fats and fatty oils by hydrolysis, and to such other organic acids as yield soaps on treatment with alkaline or basic media.

Soaps as commonly made are melted with the aid of a water content. This water content on the freshly made soap approximates thirty per cent. When such soaps are subjected to temperatures slightly in excess of 100 degrees C., the boiling point of water, they lose their water rapidly and eventually become anhydrous or substantially anhydrous. At the higher temperatures employed in my process water is even more volatile and soaps become substantially anhydrous even though it is probable that they may contain traces of water. When hereinafter I use the term "anhydrous soap" it means substantially anhydrous and free from all but traces of water.

I hereinafter in the claims intend the term "fat" to include any fats and/or fatty oils or glycerides which are useful either singly or as mixtures in the manufacture of soaps by common soap-making methods.

I hereinafter in the claims intend the term "low grade fat" to include any material containing quantities of fat and also containing such other substances as render the low grade fat objectionably odorous and/or colored or susceptible of becoming objectionably odorous and/or colored. Such low grade fats include, among others, marine animal oils or fats, certain grades of vegetable oils or fats, house grease and garbage extraction grease.

By "thorough agitation" I hereinafter mean that degree of turbulation, induced by rapid flow through a conduit or by stirring, either mechanical or by means of vapor, which is sufficient to prevent local overheating or high temperature gradients and sufficient to insure uniformity of temperature in and throughout the mass of material which is being heated by means of heat applied to surfaces with which the material is in contact.

While I have hereinbefore described in detail a limited number of forms of my invention, I do not wish to be narrowly bound by any of the quantity proportions or conditions described, but what I wish to secure by Letters Patent is the true spirit of my invention as represented by the scope of the appended claims.

What I claim as my invention is:

1. The process of making soap and glycerine which consists in heating a mixture of fat and a base to a temperature in excess of the melting point of the resulting anhydrous soap and thoroughly agitating the mixture in an atmosphere free of air while intimately contacting the mixture with a stream of water vapor.

2. The process of making soap with an unobjectionable odor and glycerine which consists in heating a mixture of low grade fat and a base to a temperature in excess of the melting point of the resulting anhydrous soap and thoroughly agitating the mixture in an atmosphere free of air, while intimately contacting the mixture with a stream of water vapor.

3. The process of making soap with an unobjectionable odor and glycerine which consists in heating a mixture of low grade fat and a base to a temperature in excess of the melting point of the resulting anhydrous soap and thoroughly agitating the mixture in an atmosphere free of air, while intimately contacting the mixture under diminished pressure with a stream of water vapor.

4. The process of making light-colored soap with an unobjectionable odor and glycerine which consists in heating a mixture of low grade fat and a base to a temperature in excess of the melting point of the resulting anhydrous soap and thoroughly agitating the mixture in an atmosphere free from air, while intimately contacting the mixture with a stream of water vapor.

5. The process of making light-colored soap with an unobjectionable odor and glycerine which consists in heating a mixture of low grade fat and a base to a temperature in excess of the melting point of the resulting anhydrous soap and thoroughly agitating the mixture in an atmosphere free from air, while intimately contacting the mixture under diminished pressure with a stream of water vapor.

6. The process of completely saponifying fat and of separating the resulting glycerine in vapor form from the soap which comprises uniformly heating a mixture of fat and an alkaline medium to a temperature in excess of the melting point of the resulting anhydrous soap while contacting the said mixture with a stream of water vapor at low pressure, excluding air, and condensing the glycerine.

7. The process of completely saponifying fat and of separating the resulting glycerine in vapor form from the soap which comprises uniformly heating a mixture of fat and an alkaline medium to a temperature above 150 degrees C. while contacting the said mixture with a stream of water vapor at low pressure, excluding air, and condensing the glycerine.

8. The process of manufacturing soap and glycerine, one from the other separated,

which consists in maintaining a mixture of fat and alkali in a molten, substantially anhydrous state until the formation of soap is substantially complete and in removing glycerine therefrom in vapor state.

9. The process of manufacturing soap containing substantially no glycerine which comprises heating a soap and glycerine mixture uniformly to a temperature in excess of its melting point when anhydrous, excluding substantially all air from the melted mixture, intimately contacting the melted mixture with a current of steam and carrying the glycerine from the melted mixture by the current of steam.

10. The process of manufacturing soap containing substantially no glycerine which comprises heating a soap and glycerine mixture uniformly to a temperature in excess of its melting point when anhydrous, excluding substantially all air from the melted mixture, intimately contacting the melted mixture with a current of steam at a pressure less than atmospheric, and carrying the glycerine from the melted mixture by the current of steam.

11. A process for the manufacture of soap and glycerine consisting in the treatment of fats or fatty oils with an alkaline medium sufficient to effect saponification, the subjecting of the composition thus formed to heat, without local over-heating, to a temperature in excess of 200 degrees C., with the aid of agitation, with the exclusion of air, with a current of steam, in a closed vessel with the aid of diminished pressure for the volatilization and recovery of the glycerine produced, and for the volatilization and removal of volatile impurities.

12. A process of making soap and glycerine consisting of mixing fats or fatty oils with an alkaline medium, heating the mixture thus formed, with the exclusion of air, with agitation, without local overheating, to a temperature in excess of the melting point of the anhydrous soap, with the employment of a current of steam, volatilizing, condensing and recovering the glycerine thus formed, and volatilizing and removing objectionable volatile impurities.

13. The process of making bland, sweet-smelling soap from fatty acids derived from marine animal oils consisting of mixing such fatty acids with an alkaline medium, heating the mixture thus formed with the exclusion of air, with agitation, without local overheating, to a temperature in excess of 250 degrees C., with the employment of a current of steam, in a closed vessel under diminished pressure, volatilizing and removing objectionable impurities.

14. The process of saponifying esters of fatty acids to produce soap and alcohol and of separating the resulting alcohol in vapor form from the soap, which comprises uniformly heating a mixture of esters of fatty

acids and an alkaline medium to a temperature in excess of the melting point of the resulting anhydrous soap while contacting said mixture with a stream of water vapor at low pressure, excluding air, thoroughly agitating the whole, and condensing the alcohol so formed.

15. The process of saponifying esters of fatty acids to produce soap and alcohol and of separating the resulting alcohol in vapor form from the soap, which comprises uniformly heating a mixture of esters of fatty acids and an alkaline medium to a temperature above 150 degrees C. while contacting said mixture with a stream of water vapor at low pressure, excluding air, thoroughly agitating the whole, and condensing the alcohol so formed.

16. The process of separating glycerine from a partially saponified and saponifying mixture of a fat and a base which consists in uniformly heating the mixture in the presence of water vapor to a temperature producing an appreciable vapor pressure of glycerine, continuously and thoroughly agitating said mixture to prevent local over-heating, excluding air, intimately contacting said mixture with a stream of water vapor, removing the mixed vapors of water and glycerine and condensing the glycerine.

17. The process of separating glycerine from a mixture of soap and glycerine which consists in heating said mixture uniformly without local overheating to a temperature producing an appreciable vapor pressure of glycerine, thoroughly agitating said mixture, excluding air, intimately contacting said mixture with a stream of water vapor, and removing the resulting mixed vapor of water and glycerine and condensing the same while carrying on the whole at diminished pressure.

18. The process of converting soap with an objectionable odor into soap without an objectionable odor which consists in heating said soap to a temperature above its melting point when anhydrous thereby driving substantially all water therefrom, agitating to maintain substantially uniform temperature throughout said soap regardless of heating means, and excluding air while contacting said soap with a flow of water vapor at a pressure low enough to prevent the existence of liquid water.

19. The process of converting soap with an objectionable odor into soap without an objectionable odor, which consists in heating said soap to a temperature in excess of 150 degrees C. thereby driving substantially all water therefrom, agitating to maintain substantially uniform temperature throughout said soap regardless of heating means, and excluding air while intimately contacting said soap with a flow of water vapor at a pressure low enough to prevent the existence of liquid water.

20. The process of making soap and glycerine which comprises the addition of successive quantities of a mixture of fat and a base to a partly filled vessel in the presence of steam within said vessel for the purpose of forming soap, applying heat to the mixture while continuously agitating the mixture to prevent local overheating, maintaining the mixture at a temperature in excess of 200 degrees C., bringing the successive additions rapidly to a temperature in excess of 200 degrees C., and removing successive and substantially corresponding quantities of resulting soap, while continuously distilling glycerine from the soap thus formed.

21. The process of making soap and glycerine which comprises the addition of successive quantities of a mixture of fat and a base to a partly filled vessel in the presence of steam within said vessel for the purpose

of forming soap, applying heat to the mixture while continuously agitating the mixture to prevent local overheating, maintaining the mixture at a temperature in excess of 200 degrees C., removing successive and substantially corresponding quantities of soap from the vessel while contacting the contents of the vessel with a stream of water vapor, while maintaining diminished pressure within the vessel.

22. The process of hydrolyzing fat which consists in mixing the fat with soap, heating the mixture to a temperature between 150 and 350 degrees C., thoroughly agitating the mixture, excluding air therefrom, and contacting the mixture with water vapor at a pressure low enough to prevent the existence of liquid water.

MARTIN HILL ITTNER.

25	90
30	95
35	100
40	105
45	110
50	115
55	120
60	125
65	130