

Jan. 4, 1949.

M. H. ITTNER

2,458,170

CONTINUOUS FAT SPLITTING

Filed Oct. 27, 1944

5 Sheets-Sheet 1

Fig. 1.

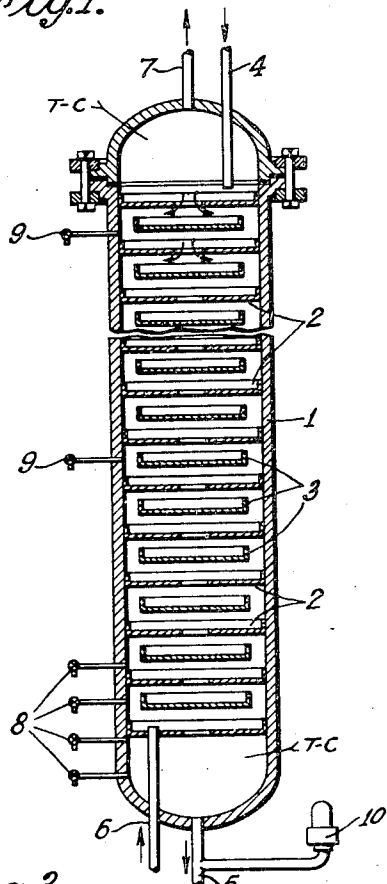


Fig. 2.

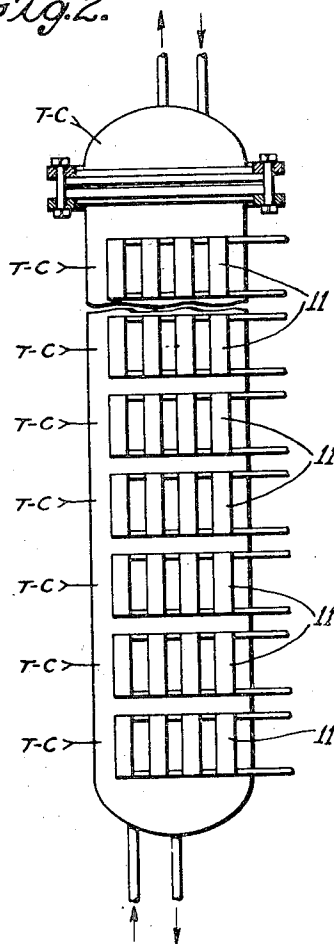
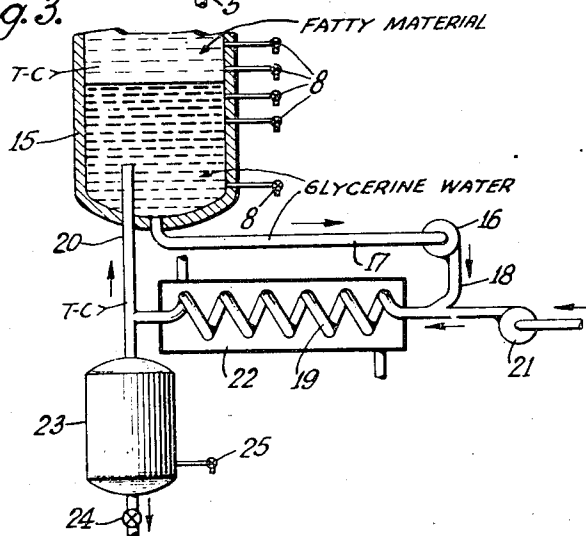


Fig. 3.



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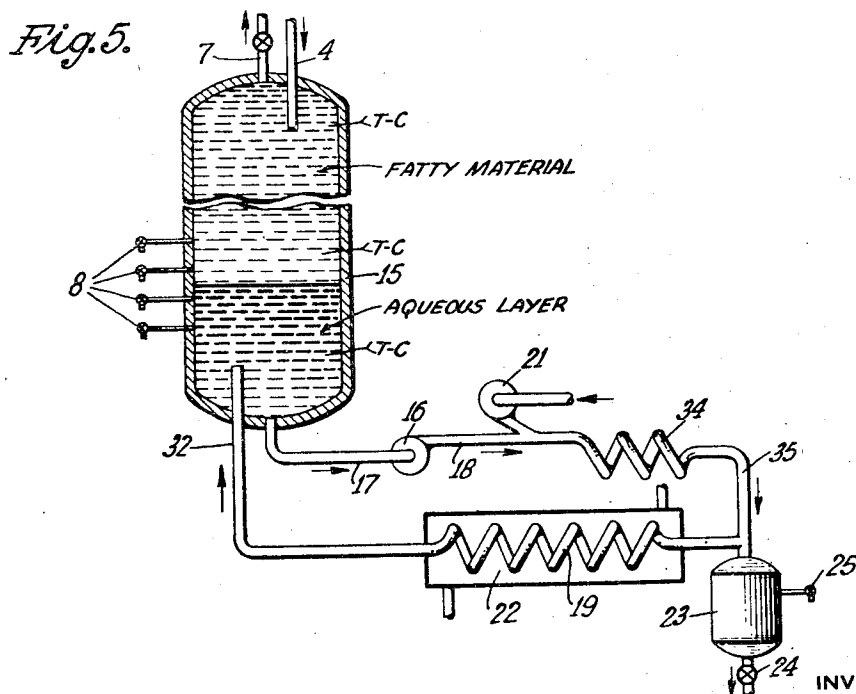
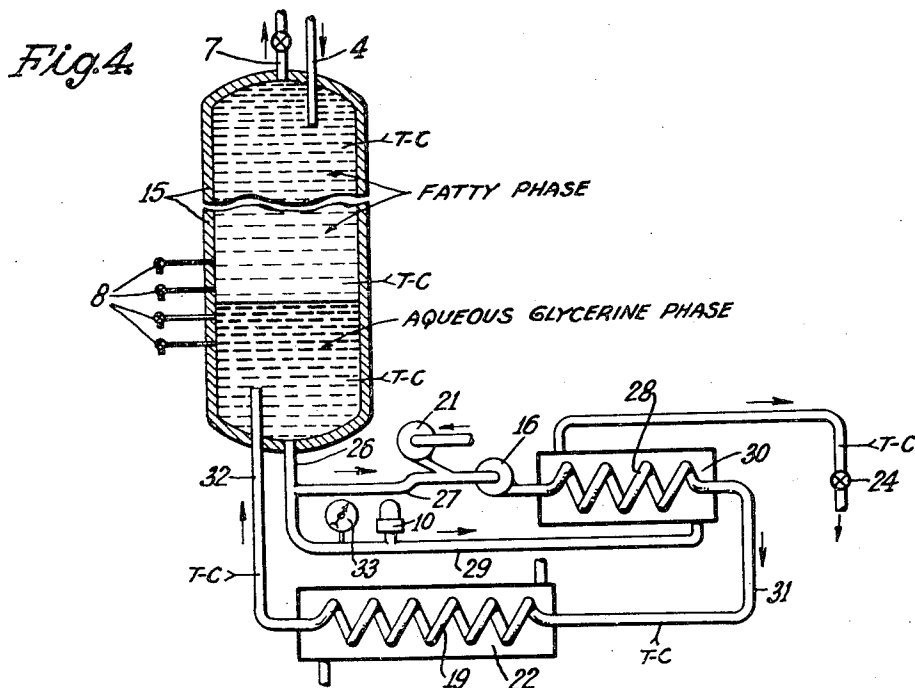
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CONTINUOUS FAT SPLITTING

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5 Sheets-Sheet 2



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CONTINUOUS FAT SPLITTING

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Fig. 6.

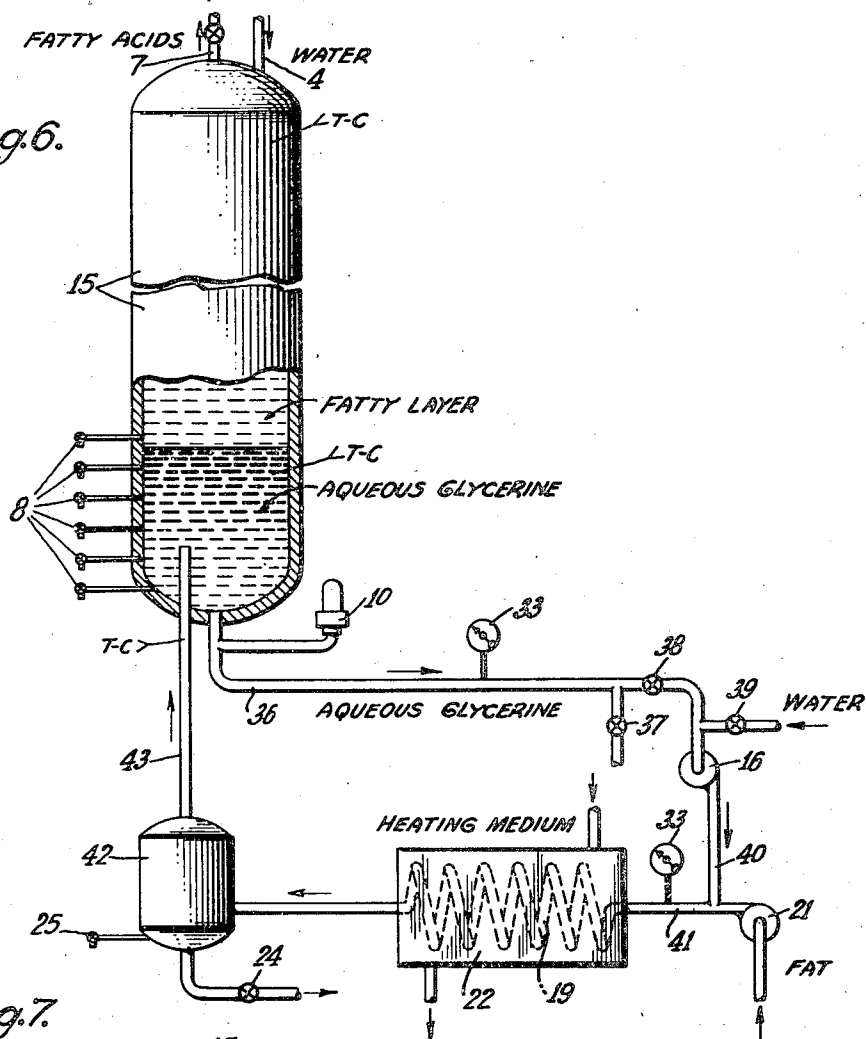
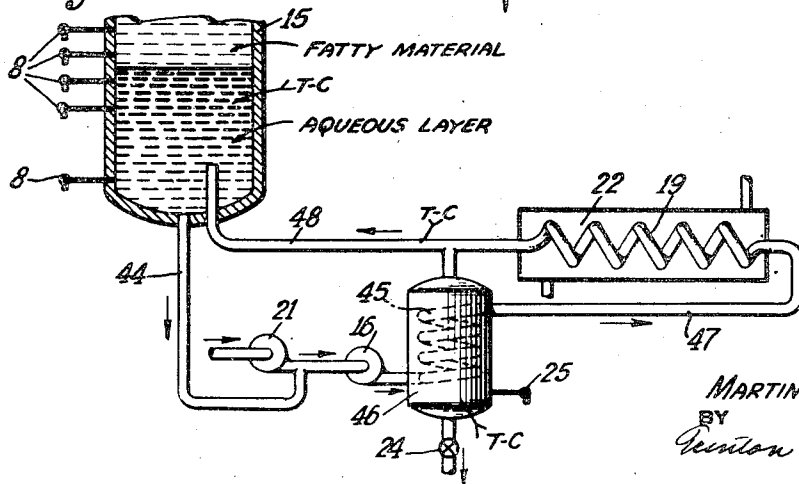


Fig. 7.



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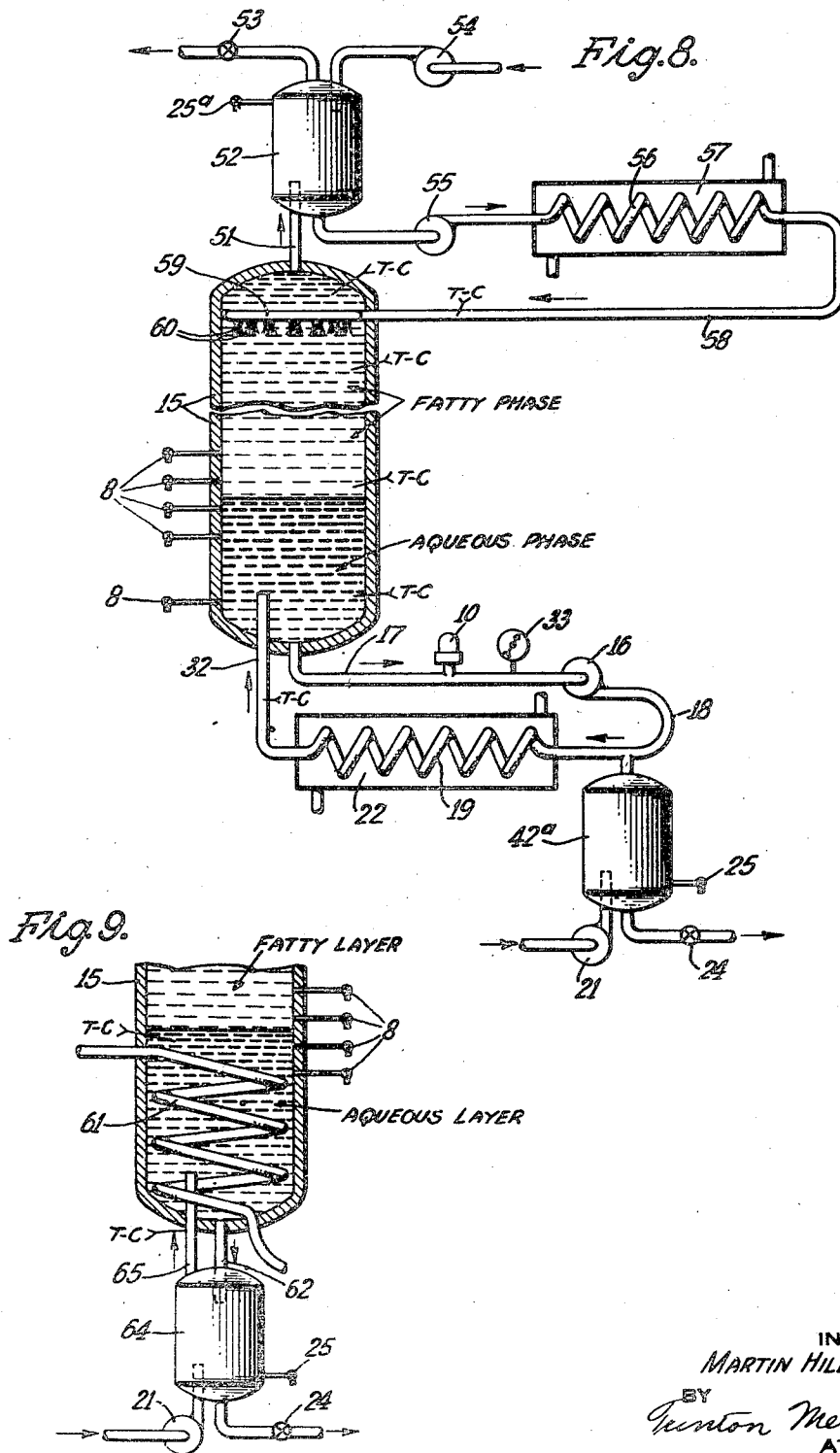
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2,458,170

CONTINUOUS FAT SPLITTING

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•5 Sheets—Sheet 4

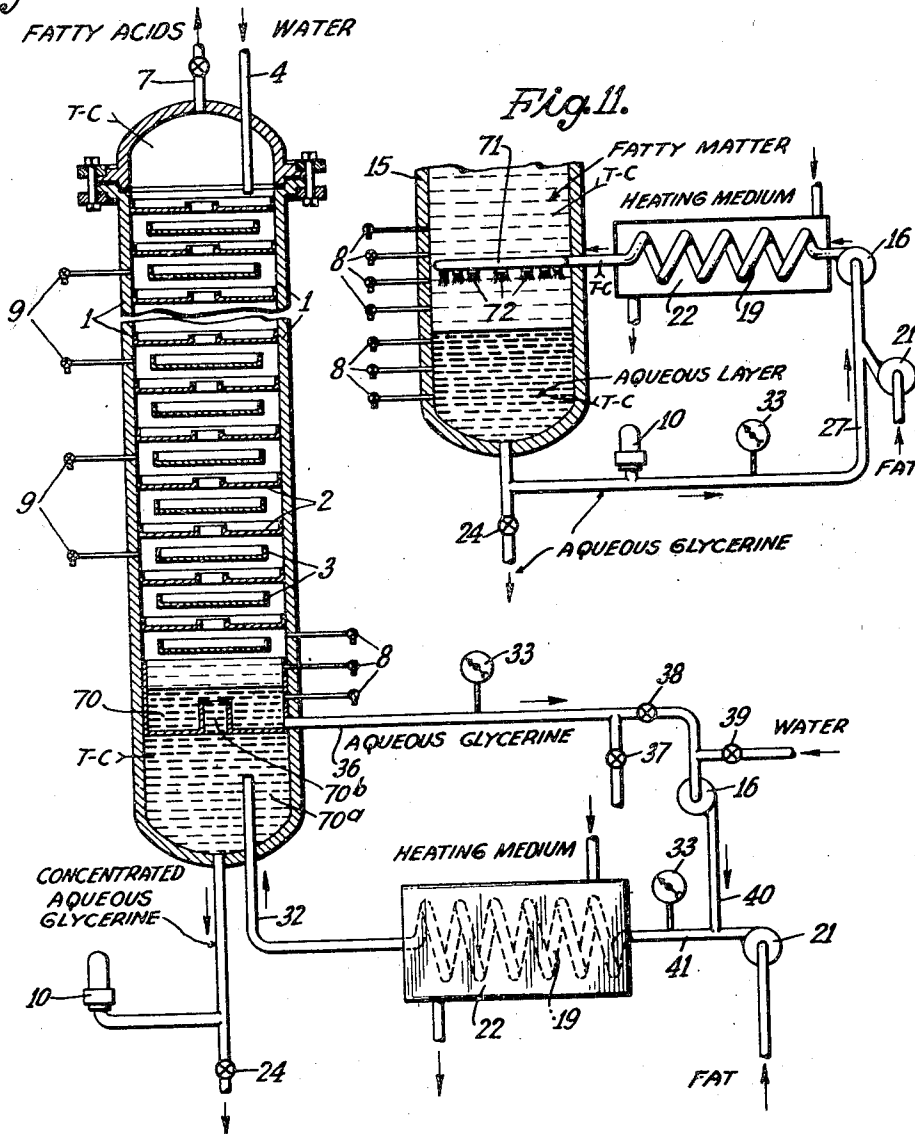


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CONTINUOUS FAT SPLITTING

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Fig. 10.



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UNITED STATES PATENT OFFICE

2,458,170

CONTINUOUS FAT SPLITTING

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Application October 27, 1944, Serial No. 560,640

19 Claims. (Cl. 260—415)

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This invention relates to an improved process for the aqueous saponification or hydrolysis of fats and fatty oils to produce fatty acids and glycerine.

Fats and fatty oils as they occur in nature consist primarily of triglycerides, that is, compounds in which one molecule of glycerine has been combined with three molecules of fatty acid with the elimination of three molecules of water. It is an object of this invention to provide an improved process for splitting or hydrolyzing such fats or oils, or triglycerides, into fatty acids and glycerine. It is another object of this invention to perform such hydrolysis with the production of a relatively concentrated aqueous solution of glycerine and relatively pure fatty acids uncontaminated by decomposition products or tars or by catalytic materials or reaction products of catalytic materials. Other objects will be apparent from the disclosures hereinafter.

The present invention pertains to counter-current hydrolysis of fats and fatty oils and provides new and advantageous means and methods of performing such hydrolysis, including new and advantageous methods of contacting and heating the water used, with fatty acids formed, so as to effect desirable heat economies and to promote the efficiency of washing out of glycerine formed during the hydrolysis; improved methods of contacting the fat to be hydrolyzed, with water or glycerine water; new and improved methods of applying and conserving heat during the process; new and improved methods of preventing or lessening darkening and deterioration, during the process, of the fatty material; and other advantages all of which will be described in greater particularity hereinafter.

The hydrolysis of fats and fatty oils to fatty acids and glycerine, by the use of water and catalysts of various types, has been known and used for a long time. Included among the catalysts which have been used are lime, magnesia, and other basic materials, and sulphuric acid and other acids. Aqueous hydrolysis with the aid of such catalysts has generally been carried out in an autoclave in the presence of liquid water and steam under moderate pressure, such as pressures from 120 to 150 pounds per square inch, while employing agitation from jet steam or mechanical stirrers to promote surface contact and emulsification between fat and water.

Another method of accomplishing the aqueous hydrolysis of fats and fatty oils is the well known Twitchell process in which the fat or fatty oil is brought into contact with water and one of

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the so-called Twitchell reagents, which are acid catalysts which possess the properties of emulsifying the fat or fatty oil and of dissolving to some extent in the fat or fatty matter at the boiling point of water.

It is well known that fats and fatty oils hydrolyze to some extent when in contact with water alone at relatively low temperatures, e. g., temperatures up to about the boiling point of water. This hydrolysis, while sufficiently fast to cause fats and fatty oils stored in the presence of moisture to deteriorate, is too slow to be of value for the commercial hydrolysis of fats and fatty oils to produce fatty acids and glycerine.

It has also been proposed to accomplish the hydrolysis of fats and fatty oils by heating the fats or fatty oils together with liquid water at comparatively high temperatures, and at pressures sufficient to prevent the formation of steam in contact with the mixture being heated, with the production of an aqueous solution of glycerine and fatty acids.

All such procedures which have been used or proposed heretofore result in balanced reactions which go to an equilibrium, and the saponification or hydrolysis, as the case may be, is incomplete except with repetitions of the treatment. Except under very favorable conditions, the saponification or hydrolysis seldom proceeds as far as 90% completion. By removing the aqueous solution of glycerine formed and subjecting the fatty matter to repetitions of the treatment, the saponification or hydrolysis may be brought up to 95% of completion, or, under favorable conditions, somewhat higher. Furthermore, agitation and protracted treatment are required so to complete the reaction, and the heat losses which take place because of the repetition of the treatment and the length of the treatment become excessive. If water is not used freely in carrying out the reaction, the saponification or hydrolysis is retarded; and if water is used freely, or if many changes of water are used, the glycerine obtained is objectionably diluted and expensive to evaporate and concentrate. Also, where the treatment is prolonged or repeated, there is an increase in the darkening of the fatty acids and the formation of undesirable tars. This is particularly true in the processes in which catalysts are used.

Where catalysts are used to hasten the saponification or hydrolysis, the products produced are contaminated by the catalysts and special treatment is required to remove the catalysts.

As an illustration of the fact that these hy-

drolyses or saponifications as commonly carried out are balanced or reversible reactions which do not go to completion even at high temperatures and pressures, I have determined that if a mixture containing 35 parts of pure neutral tallow and 25 parts of pure water is heated under pressure in an autoclave to about 255° C. for about three hours, or until an equilibrium is reached, a product is obtained which is about 92% hydrolyzed. If a mixture containing glycerine and pure tallow fatty acids, in the exact amounts which would be obtained by complete hydrolysis of 35 parts of pure neutral tallow, and sufficient water to make the total 60 parts, is heated in an autoclave to 255° C. for three hours and at the same pressure as employed in the test described above, an esterification takes place with the formation of glycerides and with the production of a mixture about 92% hydrolyzed, or about 8% esterified. Thus the same equilibrium is arrived at whether neutral tallow and water are heated together with resulting hydrolysis, or whether tallow fatty acids, glycerine and water are heated together with resulting esterification. Similar experiments with coconut oil indicate that the same phenomenon occurs, except that a larger proportion of water is required for the same degree of hydrolysis. At lower temperatures and pressures, longer periods are required for the reaction to reach an equilibrium point, and at equilibrium the hydrolysis is considerably less.

The concurrent hydrolysis of fats in heated tubes, which is well known, is equivalent in result to batch hydrolysis in an autoclave inasmuch as the glycerine formed goes into solution in the water at the points where the glycerine and fatty acids are set free, and the glycerine water and fatty matter moving along together in the tubes come to a final equilibrium depending on the concentration of the glycerine solution, the proportion of fatty acids and ester remaining, and the temperature and pressure of operation.

The fact that this reaction is one which proceeds to an equilibrium, and not to completion, is in accordance with the well known law of mass action. Furthermore, as is well known, the use of catalysts in speeding up the reaction does not affect the final equilibrium reached, but merely causes the reaction to reach this equilibrium in a shorter period of time.

By the process of the present invention, I provide for the substantially complete aqueous hydrolysis of fats and fatty oils in a single operation and within a relatively short time and with marked heat economy, with the production of relatively concentrated aqueous glycerine and fatty acids uncontaminated by catalysts or other objectionable impurities requiring special treatment for their removal, and without the objectionable decomposition and formation of tars heretofore frequently encountered when carrying the hydrolysis even to a lesser extent than that to which I carry it.

I have found that, although the solubility of water in fats and fatty acids is very slight at low temperatures, and at temperatures as high as the boiling point of water at atmospheric pressure or even considerably higher, the solubility of water in fats and fatty oils and fatty acids, under sufficient pressure to prevent the vaporization of the water, increases slowly up to about 200° C. and then increases more rapidly at higher temperatures. In all cases, to dissolve any con-

siderable amount of water in the fats or fatty acids a pressure must be used sufficient to maintain a liquid aqueous phase in contact with the fats or fatty acids, or a pressure above about the pressure of saturated water vapor at the temperature selected. As examples of the solubility of water in fatty acids: At a temperature of 235° C. and an absolute pressure of 700 pounds per square inch, fatty acids from coconut oil will dissolve about 17% of their weight of water in the presence of excess liquid water. The same fatty acids, in the presence of excess liquid water and at a temperature of 245° C. and an absolute pressure of 800 pounds per square inch, dissolve about 20% of water. At a temperature of about 287° C. and a pressure in excess of 70 atmospheres, these fatty acids blend freely with more than their own weight of water with the formation of a single, clear liquid phase. Other commercial fatty acids, such as those from tallow and palm oil, behave in a similar manner and have an increased power to dissolve water at elevated temperatures and pressures such as those above referred to.

In accordance with the present invention, I subject the fat, fatty oil, or mixtures thereof to be hydrolyzed to the action of liquid water at high temperatures and pressures. The process employs the contacting of fatty material with the water by countercurrent flow under such conditions as to insure at all times intimate contact of the fatty material with the water, with prevention or minimizing of emulsification during such countercurrent flow, and with the use of temperatures and pressures sufficiently high to insure that a substantial amount of water will actually dissolve in the fatty material, thus supplying, where and when it is needed, the water that enters into the chemical reaction of the hydrolysis. For example, coconut oil for complete hydrolysis requires about 8.3% of its own weight of water, and tallow requires about 6.4% of its own weight of water. For this rapid hydrolysis of these fats, the most effective conditions of temperature and pressure are such that the fatty material dissolves considerably more water than these amounts. The water dissolved in the fat affects the saponification, setting glycerine free, and any excess water not dissolved washes out glycerine by countercurrent washing so that the saponification may thus be carried to completion. The water thus employed for countercurrent washing dissolves glycerine in increasing amounts as it flows downwardly through the fatty material from the point of introduction to the point where it is separated from the fatty material as an aqueous glycerine solution. The water that is brought into contact with the fatty material may thus contain more or less glycerine in solution, depending upon the position in the column and the operating conditions. The word "water" is used in the specification and claims in this sense, i. e., "water" is used in the generic sense to embrace water with or without other materials present therein.

The total amount of water used in carrying out my process must be sufficient not only to provide the amount of water chemically necessary for the hydrolysis, and any excess water dissolved in the fatty material at the high temperatures and pressure employed, but also to dissolve out the glycerine formed and remove it from the fatty material by countercurrent action as an aqueous solution. By my process, I can obtain glycerine in aqueous solutions of concentrations ranging

from 15% or less to 50% or somewhat more, directly, and the amount of water used in carrying out my process should be such as to give a glycerine solution of the desired concentration.

In carrying out this process, with the aid of countercurrent flow of the fatty material to be saponified and the water, it is essential that the pressure maintained in the apparatus shall be sufficient so that even at the highest temperature selected, ordinary variations in temperature and pressure cannot result in any substantial vaporization of water within either the fatty phase or the aqueous phase to interfere with the operation of the process.

It is important, in carrying out the process, to provide for the proper separation of the aqueous glycerine from the fatty material at the point where the aqueous glycerine is removed from the apparatus, and to provide for the proper separation of the fatty material, that is, the fatty acids resulting from the hydrolysis, from the water at the point where the fatty acids are withdrawn from the apparatus. This is accomplished by providing settling spaces or zones adjacent to the parts of the apparatus at which these materials are removed, so that where the aqueous glycerine is separated or removed from the apparatus, there is provided a quiescent zone in which the aqueous glycerine may separate from the fatty material to permit the removal of aqueous glycerine free from fatty material except in so far as the fatty material may be dissolved in the aqueous glycerine. Otherwise, the incoming fatty material will readily emulsify with and be largely removed by the outwardly flowing current of aqueous glycerine when the equipment is being used at an economic rate of operation, as the fatty material is very prone to objectionable emulsification in its early stages of hydrolysis. Similarly, at the point where the fatty acids are removed from the apparatus, there is provided a quiescent zone where the fatty acids may separate from the water and be removed without removing water at the same time, except in so far as the water may be dissolved in the fatty material. Because of the fact that the temperatures and pressures are sufficiently high in my process to cause a very considerable amount of water actually to dissolve in the fatty matter, agitation is not needed to promote saponification. Agitation promotes emulsification that is generally considered as desirable, if not necessary, to increase contact in other methods employing temperatures and pressures not sufficiently high to cause a considerable degree of solubility of water in the fatty material. Emulsification ordinarily lessens the efficiency of countercurrent hydrolysis, and emulsification of inflowing fat with outflowing aqueous glycerine or of outflowing fatty acids with inflowing water, besides disturbing the smooth operation of the process, also introduces an indefinite and irregular factor in the proportions of reacting materials remaining in the apparatus.

While the process can be carried out over a fairly wide range of temperatures, which varies with different fatty materials, and over a fairly wide range of pressures, which more or less depends upon the temperatures selected, the temperatures used, and the corresponding pressures used, must be such that two separate and distinct liquid phases are maintained within the apparatus, one phase being the fatty material, that is, the fat or fatty oil, or the fatty acids, or partially hydrolyzed fatty material, containing a substan-

tial amount of dissolved water which under the most favorable conditions of operation is somewhat greater than the amount of water required for the complete hydrolysis of the fatty material, and the other liquid phase consisting of water, or aqueous glycerine, sufficient in amount effectively to wash out the glycerine from the fatty material and carry it through the apparatus, to be removed as aqueous glycerine ranging in concentrations from 15% or less up to 50% or more. Stirring or emulsification at any point may hamper the washing at that point by preventing the ready separation of the aqueous glycerine phase from the fatty phase and efficient washing out of glycerine. The pressure selected depends upon the temperature and, in order to favor solution of water in the fatty material, must be sufficient to maintain the materials in a liquid state with no substantial vaporization, within either the fatty phase or the aqueous glycerine phase, even should a slight drop in pressure or increase in temperature take place.

While no definite temperature limits can be given for carrying out the process, inasmuch as the temperatures vary with different fats or fatty oils, I have found that coconut oil, for example, can be advantageously treated at temperatures of around 235° C. or 245° C. and at pressures somewhat in excess of the pressure of saturated steam at these temperatures, and that other fats and fatty oils may be advantageously treated at similar temperatures and approximately similar excess pressures. At temperatures of about 287° C. or higher and correspondingly high pressures, I find that the process cannot be carried out with coconut oil, as at these high temperatures and pressures coconut oil fatty acids blend freely with more than their own weight of water to form a single clear liquid phase, instead of the two liquid phases required for the proper operation of my process. Similarly, with each fat or fatty oil, there is an upper temperature limit, approaching which the solubility of the water in the fatty material increases so rapidly and to such a degree as to be unfavorable to carrying out the process.

In general, I find that temperatures somewhat above about 200° C. or a little below 200° C. are satisfactory, in that at such temperatures the solubility of the water in the fatty material is quite appreciable and is in excess of the amount required for the hydrolysis of the fat or fatty oil, but is not so great as to cause the formation of a single liquid phase in the apparatus or to render the water so soluble in the fatty material as to preclude the proper washing out of the glycerine, at least with a reasonable amount of water. At substantially lower temperatures, water is not sufficiently soluble in the fatty material to allow the process to be economically carried out, and the desired hydrolysis cannot be obtained at such lower temperatures in apparatus which does not depend upon mixing and large surface contact, at least in a reasonably short time and with the production of concentrated aqueous glycerine substantially free from fatty material.

Apparatus suitable for the practice of the process of the invention comprises one or more suitable pressure vessels made of stainless steel or other suitable material having the necessary strength and resistance to the corrosive action of fatty acids and water at high temperatures and pressures and of sufficient strength to withstand the high pressures used with an ample margin of

safety. The arrangement of the apparatus is such that the fatty material and water or aqueous glycerine, which flow countercurrently, are in intimate contact over a relatively long path and do not have a tendency to emulsify to any great extent. It is advantageous to cause the fatty material to flow upwardly through the vessel or vessels while the water or aqueous glycerine flows downwardly in intimate contact with it. Where a single pressure vessel is used, it may be provided on the inside with a number of trays or other suitable devices to insure intimate contact of the fatty material and the water or aqueous glycerine, and to insure that this intimate contact is sufficiently prolonged to provide the desirable washing out of glycerine from the fatty material which leads to the substantially complete hydrolysis of the present invention. Where a plurality of pressure vessels is used, or a single pressure vessel of considerable height, the trays or other devices are not necessary, as the length of the path over which the materials flow in contact is sufficient to bring about the necessary intimacy and length of path and time of contact, but trays or similar devices may be provided with advantage if desired.

The apparatus may also be provided with suitable heat exchangers for recovering heat from the outgoing products that may be utilized elsewhere in the process, or for other purposes. Devices are also furnished to supply heat to ingoing materials, part of which may be supplied by heat recovered from outgoing material by direct or indirect heat exchange, and part of which may be furnished by diphenyl heaters, or high pressure steam heaters, or other suitable heating means. Suitable unitary heating units, such as electrical heaters, may be properly placed around the apparatus so as to supply heat to the contents, in special locations where it is most desirable to do so. Heat may be supplied at any part of the apparatus to make up heat lost by radiation, or for other purposes. The apparatus is also provided with efficient insulation to minimize heat losses.

The apparatus is also suitably provided with pumps to introduce, circulate and mix the water and fatty oil or fat as desired under the high pressure used, and with suitable gauges and thermometers and suitable safety devices such as safety valves and the like to control the pressure and temperature within the apparatus.

It is preferable to maintain within the apparatus a larger upper layer of the lighter fatty material and to maintain below the fatty material a settling zone for the separation of the aqueous solution from the fatty material. The apparatus is therefore provided with a suitable means of determining the position of the interface between the fatty material and the settling zone containing the quiescent aqueous glycerine, near the point where the aqueous glycerine is removed and near the point where the fat or fatty oil is introduced. The determination of the location of the oil or fat and water interface may be accomplished by means of a gauge glass suitably located or by providing a number of small valves or outlets in vertical series near the level at which it is desirable to maintain such an interface.

The present invention pertains to improvements in countercurrent hydrolysis, whether such countercurrent hydrolysis may be considered "continuous," "intermittent" or "alternate." In

any case, the type of process under consideration involves continuity of operation as a whole, with continuity of the parts thereof, or a continuity of series of operations following one another with regularity at short intervals. Such operations, at intervals, in series, may involve mere unintentional pauses due to pulsations of reciprocating pumps, or intentional sensible pauses in the flow of any of the materials, without affecting the general continuous character of the hydrolysis.

Fats and fatty matter when heated even to the temperature of the boiling point of water, with exposure to air, suffer some darkening and deterioration. With some fats this objectionable action is comparatively slow while with others it is quite rapid. With all fats this darkening and deterioration is seriously hastened with increasing temperatures much above the boiling point of water.

I have found that substantially all fats and fatty oils, even with the practical exclusion of air, suffer some darkening and deterioration where heated by themselves for any appreciable length of time to temperatures suitable for the process of countercurrent hydrolysis of fats. The rapidity of this darkening and deterioration is greater with some fats than with others and in all cases it is greatly hastened as temperatures substantially above 200° C. are employed, and especially at temperatures at which the countercurrent hydrolysis of fats becomes very rapid as for instance at about 240° C. or 250° C.

I have also found that this tendency of fats to darken or deteriorate may be minimized, or in some cases even avoided to all practical purposes, if the heating of the fat is accomplished in the presence of water, or water containing glycerine in solution.

In processes that involve the heating of fats, which are operated commercially and economically at high capacity, it is customary and even necessary to employ heating surfaces, the temperatures of which are very substantially above the temperature to which it is desirable to heat the fat, to provide flow of heat from the heating surface to the fat. In such equipment, when the fat is heated alone, there is always a film of fat in contact with the highly heated heating surface which becomes heated approximately to the temperature of the heating surface and which is thus exposed to conditions promoting serious darkening and deterioration.

In my preferred method of heating fats for counter-current hydrolysis I have found a way of avoiding this highly heated film of fat which is necessarily produced when the fats are heated alone in dry condition, and am thus enabled to avoid the darkening and deterioration that will otherwise occur. I rapidly circulate a large volume of water containing dissolved glycerine, drawn from or near the bottom of the pressure vessel in which the countercurrent hydrolysis is performed, through a heating zone where heat may be applied indirectly in desired quantities in any suitable manner, redischarging the reheated glycerine water back into the pressure vessel at a point appreciably above the bottom of the vessel. This may all be done without substantial loss of heat and with the employment of very little power, as the equipment is provided with heat insulation and the circulating pump discharges under substantially the same pressure head as that maintained on its intake side. I prefer to force the fat to be heated before it is sub-

jected to countercurrent hydrolysis, in desired amounts, directly into the rapidly circulating glycerine water before passing the latter through the indirect heater. In this way the fat mixed with a large volume of water or glycerine water passes rapidly through the heater without any portion of it being subjected to any substantial degree to overheating and consequent darkening and deterioration, while it is at the same time always under the salutary protection of the water, or glycerine water, with which it is mixed.

In the practice of the present invention, where a large volume of water, or glycerine water, is passed through the indirect heating zone, the heating surfaces are at all times substantially completely wetted by and in contact with water or glycerine water with the admixed fat suspended therein. It may be seen on a little consideration that the heating of the water, or glycerine water, is indirect from the surfaces of the heater, but that the heating of the fat is substantially entirely direct, the fat in finely suspended form deriving its heat in greater part, if not entirely, directly from the hot water in which it is mixed. This is a very much safer and superior way of heating the fat and completely avoids any overheating thereof.

At the point of mixing the cooler fat into the hot glycerine water there is a direct heating of the former and cooling of the latter without sensible loss of heat from the process, and the indirect heat that is applied to the mixture, regardless of the point of mixing, should be just sufficient to bring the temperature of the mixture when forced into the autoclave to the temperature at which it is desirable to start the countercurrent hydrolysis of the fat. In general this is somewhat above about 185° C.

The method of the present invention must not be confused with my concurrent-countercurrent method of hydrolysis, U. S. patent application Serial No. 138,311, filed April 22, 1937, now U. S. Patent No. 2,221,799, as in that process the temperature and length of time of concurrently heating fat and water, or fat and glycerine water, are intentionally sufficiently high and sufficiently prolonged so that a substantial degree of hydrolysis takes place during the concurrent heating part of the process. On the other hand, the method of the present invention derives its principal advantage from the rapid heating of the fat by and in the presence of a large volume of rapidly circulating water, with substantially entire absence of overheating, the heating being accomplished to the desired temperature for countercurrent hydrolysis without the fat undergoing any substantial degree of hydrolysis such as is an essential part of my concurrent-countercurrent process.

The present invention possesses a further advantage over other countercurrent methods of hydrolysis in that the method employed herein of heating the fat while intimately mixed with and finely suspended in glycerine water results in substantially saturating the fat with dissolved water by extracting this water from the glycerine water solution, thus increasing the concentration thereof. This advantage will be at once apparent if this method is compared with one employing the discharge of the hot dry fat into the pressure vessel at the level of the interface between the fatty phase and the aqueous phase, just at the bottom of the fatty phase, where it can extract little or no water from the glycerine water solution with which it scarcely comes in contact, thus effecting little if any concentration thereof,

but gradually, in the rise of the fatty layer, taking up the water, until it becomes saturated, from the washing water falling in finely divided form through the fatty layer, and thus lessening in some degree the washing effect so essential to complete hydrolysis.

The process of the invention is not confined to a single type of apparatus but may be carried out in a considerable variety of apparatus which lend themselves to the performance of the essential principles of the invention.

A clearer understanding of the invention may be obtained by a study of the accompanying drawings which illustrate various methods of applying the invention and suggest others covered by the same general principles herein described.

Fig. 1 represents in section and partly cut away one form of apparatus suitable for carrying out countercurrent hydrolysis of fats with certain supplemental equipment not shown.

Fig. 2 represents one method of applying unitary heaters to different zones of an apparatus such as illustrated in Fig. 1.

Figs. 3, 4, 5, 6, 7, 8, 9, 10 and 11 show diagrammatically different arrangements of apparatus suitable for carrying out the various processes of the invention.

In Fig. 1, 1 represents a suitable pressure resistant and corrosion resistant vertical vessel conventionally shown. It may be, for example, two feet in diameter and fifteen feet high. It is supplied with a plurality of trays 2 and 3 so as to provide a longer labyrinthian path for the flow of material. The trays may be dispensed with, especially if the height of the vessel is substantially increased, as for example, to thirty feet or more. An inlet 4 for water under pressure is provided near the top, and an outlet 5 for glycerine water under pressure is provided at the bottom. An inlet 6 is provided near the bottom for a heated mixture of fat and glycerine water under pressure, and an outlet 7 is provided at the top for fatty acids under pressure. Cocks 8 suitably placed serve as a means for locating the interface between the fatty phase and the aqueous phase and cocks 9 permit the drawing of samples from various parts of the vessel as desired. Thermocouples T-C are provided to indicate temperatures maintained in various parts of the apparatus. A safety pressure relief valve 10 is also provided.

Fig. 2 represents one means of providing separately controlled unitary heaters 11 for the separate or joint heating of the various zones of the apparatus, as for example, as illustrated in Fig. 1. Suitably placed thermocouples T-C act as indicators of the temperature maintained in various zones of the apparatus and guides for the heating which may be automatically controlled and may be applied electrically, or by other means.

Fig. 3 represents the lower part of an apparatus suitable for countercurrent hydrolysis of fats by means of the invention. The lower part of pressure vessel 15 without the use of trays is shown partly cut away and partly in section, with the glycerine-water phase and lower part of the fatty phase showing. Glycerine water is circulated rapidly and in considerable quantity by means of pump 16 from the bottom of the pressure vessel 15 through pipes 17 and 18 and heating coil 19 through pipe 20 back to pressure vessel 15 at a point somewhat above the bottom thereof. Fresh fat is injected by pump 21 into the stream of hot glycerine water just before it passes through heating coil 19 and at once becomes suspended

therein and mixed therewith and the two become heated together in passing through coil 19 of heater 22 and pass together heated to the desired temperature through pipe 20 into the lower part of pressure vessel 15 somewhat above the bottom thereof. As the glycerine water is quite appreciably heavier than the fat, a sufficient portion of it drops through the lower part of pipe 20 into small settling vessel 23 so as to keep 23 substantially full of glycerine water at all times, substantially all the fat passing from heating coil 19 directly through the upper part of pipe 20, together with a substantial quantity of glycerine water, into vessel 15. Glycerine water is drawn out of vessel 23 through pressure relief valve 24 at the required rate to maintain the interface between the fatty matter and glycerine water in vessel 15 at the desired position as may be indicated by cocks 8. A small cock 25 attached to vessel 23 will show if the glycerine water is separating properly from the fat. Thermocouples T-C for indicating temperature are installed wherever it is desirable to measure the temperature.

Fig. 4 represents diagrammatically apparatus for carrying out the invention while recovering some of the heat of the outgoing hot glycerine water to heat the incoming fresh fat after mixing it with a portion of the glycerine water. The pressure vessel 15 which is without trays is shown partly in section, partly cut away, with the fatty phase and aqueous glycerine phase and interface between them. The operation of the apparatus is as follows: Circulating pump 16 draws glycerine water freely from the bottom of the vessel 15 through pipes 26 and 27 and forces it through coil 28. Pump 21 forces fresh fat into the current of glycerine water passing through pipe 27 and the mixture of fat and water is passed through pump 16 and coil 28, located in heat exchanger 30, where the mixture of glycerine water and fresh fat takes up some heat by indirect heat exchange from a portion of the hot glycerine water from the bottom of vessel 15 passing through pipes 26 and 29 and countercurrently through the shell of heat exchanger 30. The portion of glycerine water which has thus been partly cooled in heat exchanger 30, by giving heat to the mixture of fat and glycerine water, is drawn from pressure relief valve 24 at the proper rate to maintain the interface between the fatty matter and aqueous phase in vessel 15 at the desired location, and the glycerine is recovered from the glycerine water thus obtained. The mixture of fat and glycerine water partly heated in coil 28 of heat exchanger 30 passes through pipe 31 and heater coil 19, in heater 22, through pipe 32 into the pressure vessel 15 somewhat above the bottom thereof. The equipment is provided with pressure indicator 33 and safety pressure relief valve 10 as well as thermocouples T-C as needed.

Fig. 5 represents another satisfactory arrangement of apparatus for carrying out the invention. Circulating pump 16 draws glycerine water from the bottom of vessel 15 through pipe 17 and forces it into pipe 18 where it is joined by the supply of fresh fat forced into pipe 18 by pump 21. The mixture of fresh fat and glycerine water is forced by pressure from pumps 16 and 21 through mixing coil 34, pipe 35 and heating coil 19 by way of pipe 32 into vessel 15 somewhat above the bottom thereof. The withdrawal of glycerine water as desired through separator 23 and pressure relief valve 24 is similar to that already described in connection with similar parts in Fig. 3.

Fig. 6 represents another arrangement of ap-

paratus for carrying out the invention. A pressure vessel 15 suitable for carrying out countercurrent hydrolysis is shown partly in section and partly cut away. Valves 37 and 39 being closed and valve 38 open, glycerine water solution from the bottom of vessel 15 is drawn through pipe 38 and circulated rapidly by pump 16 through pipes 40 and 41 and heating coil 19, through small separating vessel 42 and pipe 43, back to vessel 15 somewhat above the bottom thereof. Fresh fat is forced by pump 21 into the stream of water or glycerine water in pipe 41 and circulates therewith through heating coil 19 and connections until the mixture of heated fat and water is discharged into vessel 15. Separating vessel 42 is filled mainly with glycerine water, the fat rising rapidly through it and pipe 43 to vessel 15 as fast as it emerges from the heating coil 19. The lower part of separating vessel 42 contains glycerine water substantially free from fat and the glycerine water may be drawn off through valve 24 at a desired rate or so as to maintain the proper progress of the process. Pressure gauges 33 are supplied as needed and thermocouples T-C are provided at all parts where it is desirable to measure temperatures. If desirable to do so, glycerine water may be drawn off through valve 37 by opening same and closing valve 38, and fresh water may be forced in through valve 39 by opening same.

Fig. 7 represents a different arrangement of apparatus for carrying out the process of the invention. The lower part of a pressure tank 15 for countercurrent hydrolysis is shown in section and partly cut away with the interface between the fatty phase and the aqueous glycerine layer illustrated. Glycerine water is drawn from the bottom of the apparatus through pipe 44 and meets a current of fresh fat injected into the system by pump 21. The mixture of glycerine water and fresh fat is impelled forward by circulating pump 16 through coil 45 located in the combined heat exchanger and separating vessel 46 and passes by way of pipe 47 into heating coil 19 and through pipe 48 into vessel 15 somewhat above the bottom thereof. Heat exchanger-separating vessel 46 is connected in its upper part with pipe 48 so that it is at all times substantially full of glycerine water solution, whereas substantially all the fat which has been heated in passing through coil 19 of heater 22 passes on with a portion of the glycerine water through pipe 48 into vessel 15. A certain degree of heat economy is effected in combined heat exchanger-separator 46 and this may be increased by arranging that the material passing through the coil 45 and that surrounding it pass each other countercurrently. Concentrated glycerine water, which has been somewhat cooled by imparting a portion of its heat indirectly to the fat-water mixture passing through coil 45, may be withdrawn at a desirable rate through valve 24.

Fig. 8 shows still another arrangement of apparatus suitable for carrying out the novel features of the invention. A pressure vessel 15 is shown in section, partly cut away, and is designed to operate without the use of trays. Some of the aqueous phase is drawn from the bottom of the vessel 15 through pipe 17 and is forced by pump 16 through pipe 18 and heating coil 19, in heater 22, through pipe 32 back into vessel 15 somewhat above the bottom thereof. Separating vessel 42a is connected in its upper part with pipe 18 so that the glycerine water which is being circulated through pipe 18 is free to enter vessel 42a and

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fill it. Fresh fat is forced in desired amounts and at desired rates by pump 21 into the lower part of vessel 42a and is discharged directly into the hot glycerine water in vessel 42a and rises through it, becoming gradually heated by direct contact and becoming at the same time substantially saturated with water absorbed out of the glycerine water which is thus cooled somewhat by direct countercurrent contact with the fat which is thus being heated, while at the same time increasing the glycerine content of the solution by removal of some of the water therefrom. The fat rises through vessel 42a and joins the stream of glycerine water passing through pipe 18 so rapidly that vessel 42a remains full of glycerine water except for the rising stream of fat. Glycerine water which has been somewhat cooled and somewhat concentrated by extraction of some of the water by the fat, by direct countercurrent contact with the fat, may be drawn off through valve 24 at a desired rate. Safety relief valve 10 and pressure gauge 23 are provided as a precaution against the use of excessive pressures and to indicate the actual pressure employed within vessel 15. Figure 8 also illustrates a novel and very advantageous method of removing the finished fatty acids from the upper part of vessel 15 and of imparting part of the heat of the fatty acids produced, by direct heat interchange, to the incoming fresh water, and a means of more effectively subdividing and distributing the water for washing out the glycerine formed, by countercurrent flow, all of which are part of the present invention. The fatty acids produced by hydrolysis in vessel 15, passing upwardly through pipe 51, enter and substantially fill vessel 52. The upper part of vessel 52 is provided with a quiescent zone that permits the fatty acids to drop any undissolved water, after which they may be removed at any desired rate, compatible with good operation, through valve 53. Test valve 25a permits drawing off a small sample of fatty acids at any time to test its freedom from undissolved water or for other purposes. Fresh water is forced into vessel 52 somewhat below the top thereof by means of pump 54. This water in falling through the fatty acids becomes heated by direct countercurrent contact while cooling the rising fatty acids to some degree. The fatty acids thus partly cooled have a lessened capacity to dissolve water than at the higher temperature maintained in vessel 15, and give up a portion of their dissolved water in cooling, which together with the fresh water injected by pump 54, falls to the bottom of vessel 52 tending to extract the last traces of free glycerine remaining in the fatty acids. Substantially all this water is drawn from the bottom of vessel 52, together with a considerable amount of fatty acids, by pump 55, and is forced through heating coil 56 in heater 57 and pipe 58 to manifold 59, through spray nozzles 60 located within the upper part of vessel 15, where the water mixed with fatty acids and heated to about the temperature maintained in vessel 15 is sprayed in finely divided condition and well distributed over the cross section of vessel 15. The spraying and subdivision of the water is much more efficiently accomplished when thus mixed and heated with fatty acids than is possible if the water alone were to be sprayed in. It is evident that where only a limited amount of water is used, as is necessarily the case, and the water is forced in alone, the conditions favoring good spraying are less favorable than where a greater flow of material is being forced through

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the spray nozzles 60, this greater flow embracing a considerable circulation of fatty acids mixed with the desired amount of fresh water. A desirable method of operating the equipment shown in Fig. 8 is to supply all the fresh water employed through pump 54. Thermocouples are provided to supply desired information of temperatures prevailing within various parts of the equipment.

Fig. 9 shows a simple arrangement of apparatus for carrying out the invention. The lower part of a pressure vessel 15 suitable for carrying out countercurrent hydrolysis is shown partly in section and partly cut away. A heating coil 61 is provided for supplying indirect heat to the contents of the lower portion of vessel 15. It is desirable that the heating coil shall be surrounded at all times by the aqueous layer to prevent overheating of the fat which would otherwise come into contact with the heating surface of coil 61. To this end the interface between the fatty and aqueous layers may be maintained somewhat above the top of coil 61, as illustrated, and as determined by test cocks 8. Glycerine water may flow freely from vessel 15 into vessel 64 through pipe 62 extending into vessel 64 somewhat below the top thereof. Fresh fat is forced by means of pump 21 into vessel 64 to a point somewhat above the bottom thereof and, being lighter than glycerine water, rises in finely divided condition through the glycerine water in vessel 64 becoming somewhat heated while cooling the glycerine water to some degree by direct and countercurrent interchange of heat. The fat rises rapidly through vessel 64 and through pipe 65 leading out of the upper part of vessel 64 to vessel 15 at a point somewhat above the bottom of vessel 15, where it becomes rapidly heated by direct contact with the heated glycerine water in the lower part of vessel 15 to substantially the same temperature as the glycerine water in vessel 15, this temperature being controlled by heating coil 61 and the thermocouples T-C indicating the temperature. Aqueous glycerine water somewhat cooled by direct countercurrent heat exchange, and somewhat concentrated by the extraction of some of its water by the fat in becoming heated, may be withdrawn through valve 24 at a desirable rate. Safety relief valve, pressure gauge and other necessary or desirable pieces of equipment should be provided as needed or as they may be helpful to the operation and control of the process.

Fig. 10 illustrates an apparatus suitable for carrying out the invention, which embraces a pressure vessel 1 provided with a plurality of trays 2 and 3, and other equipment in which countercurrent hydrolysis of fats may be carried out. The lower part of the pressure vessel 1 is provided with two connected chambers 70 and 70a for collecting glycerine water. Although the chambers 70 and 70a are connected by a passageway 70b, the separation of one from another is sufficient to prevent the liquor in one from mixing freely with the other. An advantageous method of operation of the equipment is to draw aqueous glycerine from chamber 70 through pipe 36 and force it by means of pump 16 through pipes 40 and 41, through coil 19 in heater 22, and through pipe 32 to chamber 70a somewhat above the bottom thereof, valve 38 being open and valves 37 and 39 being closed. Fresh fat in desired amounts at desired rates is forced by means of pump 21 into pipe 41 where it meets the current of hot aqueous glycerine passing through pipe 41 and heating coil 19, the fat and water,

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in direct contact with one another, being thus rapidly heated to the temperature at which the hydrolysis is to be performed in vessel 1, before delivery to vessel 1 through pipe 32. In the apparatus of Fig. 10 the interface between the fatty layer and the aqueous layer is held preferably at an appreciable distance above the bottom of chamber 70 so as to provide a quiescent space within the lower part of chamber 70. The height of the interface may be located by means of test cocks 8 or by other suitable means. Chamber 70 will collect the aqueous glycerine formed directly by countercurrent hydrolysis and washing and provide separation from undissolved fat therein. When this aqueous glycerine withdrawn from chamber 70 through pipe 36 is rapidly heated in heating coil 19 while it is in direct contact with finely divided fresh fat, the fat will become substantially saturated with water extracted from the aqueous glycerine, thus increasing the concentration of glycerine in the resulting aqueous glycerine which when discharged into chamber 70a will not mix freely with the diluted glycerine in chamber 70. A relatively concentrated aqueous glycerine may thus be withdrawn in desired amounts at desirable rates from chamber 70a through valve 24, and the fatty acids produced may be withdrawn through pipe 7 and a valve connected therewith. The heated fat emerging into chamber 70a from pipe 32 will rise rapidly through the hot glycerine water in chamber 70a and in passageway 70b and by the time it joins the fatty phase layer will have become substantially saturated with dissolved water drawn mainly from the glycerine water in chamber 70a thus concentrating this latter. The equipment is provided with pressure gauges 33 and devices T-C for indicating the temperature of the material undergoing treatment in the various stages, safety relief valve 10 as a precaution against the development of excessive pressure within the apparatus, insulation, and other means necessary to proper operation.

Fig. 11 illustrates still another method of carrying out the principles of the invention. The lower part of pressure vessel 15 is shown, partly in section and partly cut away, as connected at its bottom with pipe 27, circulating pump 16, heating coil 19 in heater 22, manifold 71 and spray nozzles 72. In operation, aqueous glycerine is drawn freely from the bottom of vessel 15 through pipe 27 and fresh fat in desired amounts at desired rates is forced by pump 21 to join the circulating stream of aqueous glycerine or glycerine water in pipe 27. The mixture of glycerine water with fresh fat suspended therein is forced rapidly by pump 16 through heating coil 19 in heater 22, where the fat and water in direct intimate contact with one another are rapidly heated to the temperature at which it is desired to begin the hydrolysis in vessel 15, and the mixture of heated fat and water is then forced through manifold 71 and spray jets 72 located within vessel 15, in the lower portion of the fatty matter phase where the hydrolysis of the fatty matter is in its incipency. At the time when, and at the place where, the hot mixture of fat with a large volume of glycerine water is forcibly injected through a plurality of sprays 72 in the lower part of the fatty phase, a local quasi-emulsification takes place which multiplies the speed of the first stages of the hydrolysis by instantly diluting each particle of glycerine, when and where it is set free, by mixing it with the aqueous glycerine emerging from the spray nozzles 72. Although it is to be

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emphasized that, in general, countercurrent hydrolysis is retarded by local emulsification and would be entirely prevented by complete emulsification, it should be understood that this method of carrying out the invention greatly increases the rapidity and degree of countercurrent hydrolysis realized in its first stages. By this method of operation perfect emulsification is avoided even locally and there is a continuous separation of the phases of the quasi-emulsion that keeps pace with its formation, the fatty particles gradually uniting and moving upward to join the continuous fatty phase, which is undergoing countercurrent hydrolysis, and the aqueous particles containing dissolved glycerine gradually uniting and moving downward to join the continuous aqueous glycerine phase below. The position of the interface between the fatty and aqueous layers may be located by test cocks 8 and kept at the desired level with their aid. Aqueous glycerine may be withdrawn through valve 24 at a controlled rate. Thermocouples T-C are provided where desirable or necessary to a proper control of the process, and a pressure gauge 33 is also provided. A safety relief valve 10 may be supplied. Provision is made, but not shown, for withdrawal of the fatty acids produced by the countercurrent hydrolysis and for supplying the water that performs the function of hydrolyzing the fat and washing out the glycerine formed, as well as for supplying the water dissolved by the fatty matter under the conditions employed for the hydrolysis. This dissolved water, although coming originally from the water used in the countercurrent washing of the fatty matter, is taken up by the fat during the initial heating stages and is extracted right out of the glycerine water with which the fat is in direct contact while being heated, thus further concentrating the glycerine water. The circulation of glycerine water from the bottom of vessel 15 through pump 16 and heating coil 19 is so rapid that the fat injected by pump 21 and suspended in the glycerine water is brought to temperature and forced into vessel 15 before any substantial degree of hydrolysis can take place. Although the operation of the invention in the apparatus illustrated in Fig. 11 may be as I have described it, I may also obtain good results with certain advantages if the interface between the fatty and aqueous layers, instead of being maintained as shown, somewhat below the jets 72, is maintained at their level or even somewhat above the level of these jets.

Separating vessel 23 as shown in Figs. 3 and 5 may be supplied with means for indirect cooling under pressure, not shown, to provide for the more complete separation at lowered temperatures of the fatty matter that is dissolved in the glycerine water at higher temperatures. Such a cooling-separating chamber for the removal of glycerine water should be out of the direct line of flow of hot circulating liquors. The fatty matter separated in vessel 23 may be eliminated from the vessel by its tendency to rise through the water in the pipe shown as connecting with the upper part of vessel 23, or a separate pipe may be provided connecting the upper part of vessel 23 with the rest of the apparatus in such a way that separated fatty matter will be eliminated therethrough from vessel 23 because of its lighter gravity.

In an analogous way a cooling chamber may be provided near the top of the apparatus for indirectly cooling under pressure the outgoing fatty acids, whereby they will deposit the greater part of their dissolved water which will settle by gravi-

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ty through the cooled fatty acids, and this separated water may be conducted back, still under pressure, so as to serve, after reheating, as part of the water employed for countercurrently washing out glycerine and for hydrolysis of fats. The heat removed in cooling the contents of the cooling-separating chambers may be utilized by means of suitable heat exchangers.

It will be seen from a study of the processes of the invention that where the fat which is to undergo hydrolysis is not brought up to the hydrolyzing temperature by itself in a substantially dry condition, but is heated under pressure, while in direct contact with water or water containing glycerine, to above about 185° C. by the processes of the invention, it actually extracts and dissolves from the hot water, or hot water containing glycerine, a substantial amount of water, and that it is this dissolved water which directly performs the hydrolysis, gradually setting glycerine and fatty acids free. The water which is caused to flow countercurrently past the fatty material in the performance of the process is, under these conditions, not dissolved in the fatty material, and has only an indirect influence on the hydrolysis by washing away the glycerine set free, shortly after it is set free, from the material. Thus it is only the water dissolved in the fatty material which continues directly to perform the hydrolysis.

As the processes of the invention bring the fat up to hydrolyzing temperature only while it is in direct contact with water, or water containing glycerine, and as the degree of heat thus rapidly applied is sufficient to institute the hydrolysis, the fat so heated becomes saturated with dissolved water by the time the initial heating is completed. Where this dissolved water is extracted and absorbed from water containing glycerine, the concentration of the glycerine in the glycerine solutions is thus increased. Where the initial heating is to a sufficiently high temperature, as for example, to about 240° C., and where good heat insulation is employed, no further heating may be required for completion of the process. On the other hand, for certain facts that may suffer from the employment of such temperatures it may be desirable to perform the initial heating to only about 200° C. or to even a somewhat lower temperature. In such cases it is important to be able to supply additional heat, as needed, to substantially any part of the process either to make up for heat lost by radiation or to advance the rate of hydrolysis as the process progresses. The advantage of this auxiliary heating is furthered by the fact that fatty acids in the presence of water suffer even less than glycerides so heated, and, as pointed out, much less than glycerides which are heated in the absence of water.

The present application is a continuation of my application Serial Number 311,584, filed December 29, 1939, now abandoned, which was a continuation-in-part of my application Serial Number 138,311, filed April 22, 1937, now U. S. Patent No. 2,221,799.

I claim:

1. In the process of hydrolyzing fats with water, with the aid of heat and pressure and countercurrent flow between said fats and hot water, the step comprising subjecting said fats to rapid heating to above about 185° C. while in direct contact with water containing dissolved glycerine without effecting a substantial degree of hydrolysis during said rapid heating, the pres-

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sure employed for said heating and said hydrolysis being sufficient to maintain the water containing dissolved glycerine in a liquid state at the temperature employed, said temperature being sufficiently high to cause the fats to dissolve a substantial amount of water from said aqueous glycerine solution.

2. The method of hydrolyzing fats and fatty oils with water to fatty acids and glycerine, with the aid of heat and pressure, which comprises intimately contacting such fatty material with water by countercurrent flow, subjecting the fatty acids resulting therefrom to partial cooling by direct contact and mixing with water whereby the water is heated, applying additional heat to the water while mixed with a portion of said fatty acids, and passing the water thus heated into intimate countercurrent contact with the fatty material undergoing hydrolysis, while maintaining two liquid phases, a fatty phase and an aqueous phase, whereby the fatty material undergoes substantial hydrolysis with the formation of fatty acids and glycerine.

3. In the process of hydrolyzing glycerides of the higher fatty acids with water, with the aid of countercurrent contact between the fatty material and water, the steps comprising mixing the glycerides with hot water containing glycerine in solution, under pressure, bringing the temperature of the mixed glycerides and aqueous glycerine solution rapidly to above about 185° C. under pressure, the time of said rapid heating being insufficient to effect a substantial degree of hydrolysis in said glycerides, passing the fatty matter so heated in intimate countercurrent contact with water at a temperature of about about 185° C., while maintaining a fatty phase and a liquid aqueous phase with the aid of sufficient pressure, the time of said countercurrent contact being sufficient to effect a substantial degree of hydrolysis of said glycerides into fatty acids and glycerine.

4. In the process of hydrolyzing glycerides of the higher fatty acids with water, with the aid of countercurrent contact between the fatty material and water, the steps comprising mixing the glycerides with hot water containing glycerine in solution, under pressure, bringing the temperature of the mixed glycerides and aqueous glycerine solution rapidly to about 240° C. under pressure without effecting a substantial degree of hydrolysis during said rapid heating, passing the fatty matter so heated in intimate countercurrent contact with water at a temperature of about 240° C., and employing sufficient pressure to maintain a fatty phase and a liquid aqueous phase, the time of said countercurrent contact being sufficient to effect a substantial degree of hydrolysis of said glycerides into fatty acids and glycerine.

5. In the process of hydrolyzing fats with water to fatty acids and glycerine with the aid of heat and pressure and countercurrent washing of glycerine from the resulting fatty matter with the aid of hot water, the method of finely dividing said water to facilitate said washing, which comprises mixing the water with a portion of said fatty acids and forcing the mixture of fatty acids and water under pressure, into said fatty matter, with sufficient velocity to effect a fine state of division of said water.

6. In the hydrolysis of fats with water to fatty acids and glycerine, with the aid of heat and pressure, and countercurrent washing of glycerine

from the resulting fatty matter with the aid of hot water, the steps of minimizing deterioration of the fats and resulting fatty matter caused by the application of heat thereto which comprises rapidly heating said fats to a temperature above 185° C. after mixing them under pressure with water containing dissolved glycerine, the time of said rapid heating being insufficient to effect substantial hydrolysis of said fats, and subjecting the fats so heated to countercurrent hydrolysis with hot water.

7. In the hydrolysis of fats and oils with the aid of water, heat, and pressure, and countercurrent extraction of glycerine from the hydrolyzed fatty matter by means of hot water, the steps comprising concentrating the aqueous glycerine solution resulting from said hydrolysis by rapidly heating the fats and oils about to be subjected to said hydrolysis to a temperature in excess of about 185° C. while in contact with said aqueous glycerine solution without effecting a substantial degree of hydrolysis of said fats and oils during said rapid heating, maintaining said heated aqueous glycerine solution in liquid condition by pressure, whereby water for the saturation of said fatty matter is dissolved and removed from said aqueous glycerine solution, and subjecting the fatty matter thus heated to countercurrent hydrolysis.

8. In the hydrolysis of fat with the aid of water, heat, and pressure, the steps comprising increasing the glycerine concentration of aqueous glycerine solution resulting from hydrolysis of fat by rapidly heating the fat about to be subjected to hydrolysis to a temperature in excess of about 185° C. while in contact with said aqueous glycerine solution without effecting a substantial degree of hydrolysis of said fat during said heating, maintaining said heated aqueous glycerine solution in liquid condition by pressure, whereby water for the saturation of said fatty matter is dissolved and removed from said aqueous glycerine solution separating the concentrated glycerine from the fatty matter, and subjecting the fatty matter thus heated to hydrolysis with the aid of heat and water maintained in liquid phase by pressure.

9. In the process of hydrolyzing fat with water with the aid of heat and pressure and countercurrent flow between said fat and hot water, the steps of rapidly passing said fat, under pressure, mixed with water containing glycerine in solution, through a heating zone, rapidly raising the temperature of said fat while in contact with said aqueous glycerine solution to above about 185° C. without effecting a substantial degree of hydrolysis of said fat during said rapid heating, employing sufficient pressure to maintain the aqueous glycerine solution in liquid condition, and then subjecting said heated fat to hydrolysis by countercurrent flow with hot water in liquid condition.

10. In a countercurrent process of hydrolyzing fat with the aid of liquid water above 185° C., the pressures and temperatures employed being sufficient to hold a substantial amount of water dissolved in said fat during said countercurrent hydrolysis, the steps of rapidly bringing said fat up to about said hydrolyzing temperatures and pressures while in direct contact with sufficient water substantially to saturate said fat at about said temperatures and pressures before submitting said fat to countercurrent hydrolysis without effecting a substantial degree of hydrolysis during said preliminary heating, and directly sub-

mitting said heated fat to said countercurrent hydrolysis.

11. In a countercurrent process for hydrolyzing fats with the aid of water in liquid condition at temperatures above 185° C., the method which comprises the steps of rapidly applying heat to said fats while in direct contact with water to bring the fats to about the temperature employed in said hydrolysis without effecting a substantial degree of hydrolysis of said fats during said rapid heating, and directly submitting the fats thus heated to said countercurrent hydrolysis.

12. In the countercurrent process of hydrolyzing fats with liquid water to fatty acids and glycerine at temperatures above 185° C., the steps comprising supplying variable and controlled quantities of heat to the fatty matter undergoing countercurrent hydrolysis in any of a plurality of locations, bringing the fats to be hydrolyzed while at a temperature substantially below 185° C. into direct contact with water and raising the temperature of the mixture above 185° C., and extracting glycerine freed from said fatty matter by said hydrolysis by countercurrent washing with hot water and hot glycerine water contained in a liquid aqueous phase.

13. In the process of hydrolyzing fat with water with the aid of heat and pressure and countercurrent flow between said fat and hot water, the steps comprising preheating fat in contact with water to a temperature above about 185° C., the amount of water in contact with said fat during said preheating and the time of said preheating being sufficient to saturate the fat with water at the said temperature but the time of said preheating being insufficient to produce substantial hydrolysis, and then subjecting the preheated solution of water in fat to hydrolysis by countercurrent flow with hot water in liquid condition at a temperature above about 185° C.

14. In the countercurrent process of hydrolyzing fats with water to fatty acids and glycerine at elevated temperature and pressure, the method which comprises contacting hot fatty acids produced in the countercurrent process with water at lower temperature to effect cooling of the fatty acids and heating of the water by direct heat exchange, adding heat to said water from an outside source of heat, and contacting said heated water by countercurrent flow with fatty matter to be hydrolyzed.

15. In the countercurrent process of hydrolyzing fatty material with water in liquid condition in which a column of reacting materials is maintained at hydrolyzing temperature above 185° C. under sufficient pressure to maintain water in liquid condition, the steps of introducing water into the upper portion of said column, maintaining a pool of aqueous glycerine at the bottom of said column, introducing fatty material at a temperature substantially below 185° C. into said pool of aqueous glycerine, and supplying sufficient heat locally to the materials adjacent to the bottom of said column to raise the fatty material rapidly to the hydrolyzing temperature.

16. In the countercurrent process of hydrolyzing fatty material with water in liquid condition in which a column of reacting materials is maintained at hydrolyzing temperature above 185° C. under sufficient pressure to maintain water in liquid condition, the steps of introducing water into the upper portion of said column, introducing fatty material into the lower portion of said column in a state requiring substantial heat to bring

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it to the hydrolyzing conditions and supplying sufficient heat locally to the materials in the lower portion of said column to maintain the temperature thereof substantially equal to said hydrolyzing temperature.

17. In the countercurrent process of hydrolyzing fatty material with water in liquid condition in which a column of reacting materials is maintained at a hydrolyzing temperature above 185° C. under sufficient pressure to maintain water in liquid condition, the steps of introducing water into the upper portion of said column, rapidly preheating fatty material in contact with water to substantially said hydrolyzing temperature, and introducing said preheated fatty material into the lower portion of said column.

18. In a continuous process for hydrolyzing fats by causing preheated fat and preheated water to pass in continuous countercurrent direction in intimate contact with each other at a hydrolyzing temperature above 185° C. and under sufficient pressure to maintain water in liquid phase, the step of mixing water with the fat to be hydrolyzed and rapidly preheating this mixture to approximately said hydrolyzing temperature, thus

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producing a preheated solution of water in the fat, before introducing said fat into the countercurrent hydrolysis reaction zone.

19. In a process for hydrolyzing fats by causing fat and water to flow through a hydrolyzing zone in countercurrent direction in intimate contact with each other at a hydrolyzing temperature above 185° C. and under sufficient pressure to maintain water in liquid phase, the step of substantially saturating the fat to be hydrolyzed with water at approximately said hydrolyzing temperature before introducing the fat into the countercurrent hydrolyzing zone.

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