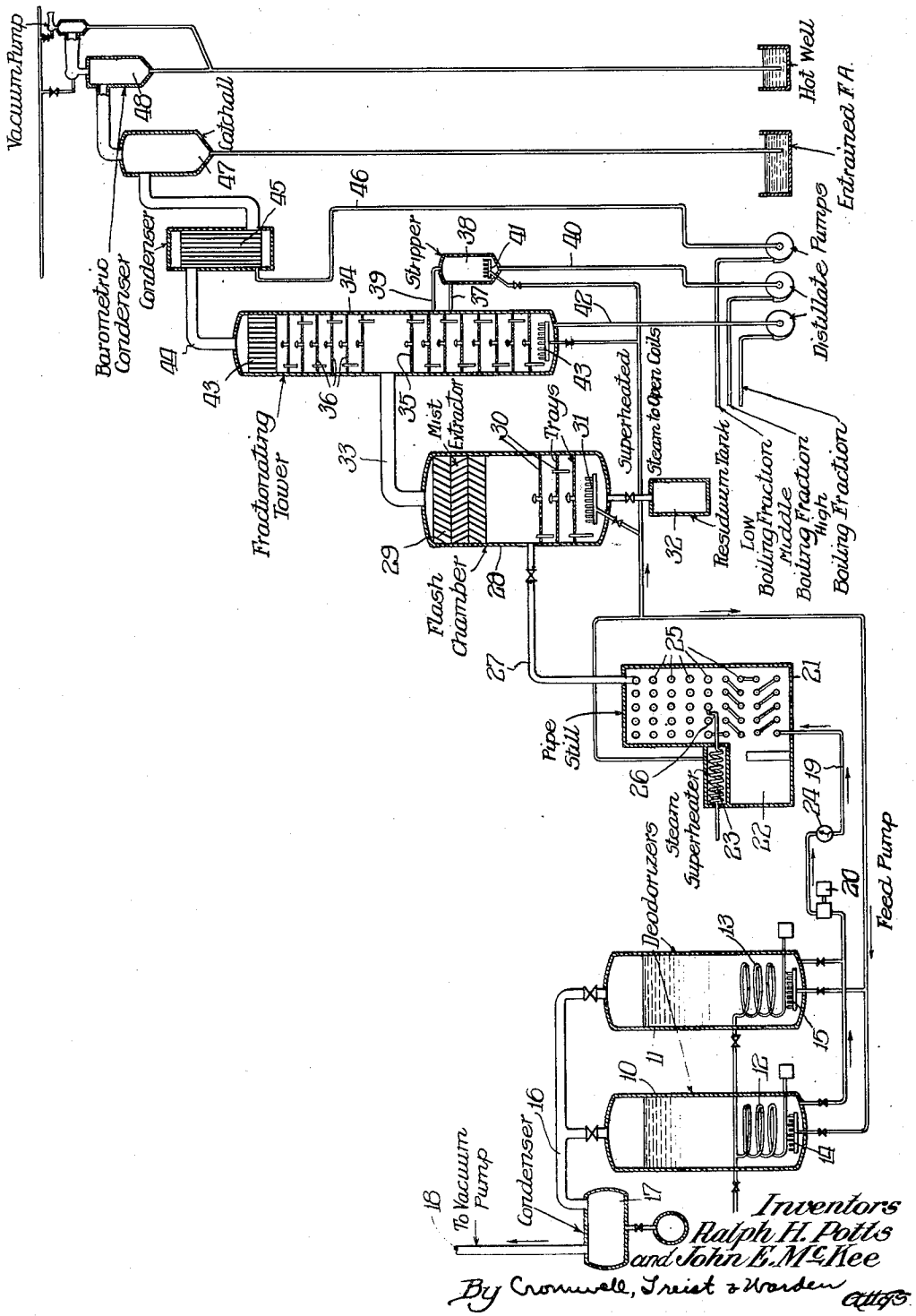
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FATTY ACID DISTILLATION

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FATTY ACID DISTILLATION

Ralph H. Potts, Chicago, and John E. McKee, Western Springs, Ill., assignors to Armour and Company, Chicago, Ill., a corporation of Illinois

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The present isvention relates to the treatment of oleaginous materials having a fatty acid content, and has particular reference to an improved process for treating fatty material obtained, for instance, from the product known as "foots" in a particularly advantageous manner.

Animal oils and vegetable oils, such as cottonseed oil, corn oil, and cocoanut oil, are customarily subjected to a process of alkali refining, whereby free fatty acids in the oil are neutralized. The resultant soap flocks to the bottom of the refining kettle, together with some neutral oil that is held by the soap particles. The material thus separating from the oil is known as "foots", and ordinarily will contain on the order of 50 per cent fatty acids, the remainder being impurities and, primarily, water. Of the fatty acid content of foots, approximately 60 per cent may be free fatty acids and the remainder glycerides.

The foots are acidulated with sulfuric acid in order to break the mass into oil and water. That is, the acid breaks the soap into fatty acid and the sulfate salt of the positive radical combined with the fatty acid. The mass then separates into an oil layer and a water layer, which layers may be separately recovered by decanting or other suitable process.

The fatty material obtained from acidulation of the foots is usually termed "raw fatty acid stock", and for purposes of description, our invention will be described in particular with reference to this material.

It has been customary heretofore to recover the fatty acids from fatty acid stock by saponifying this stock and then subjecting the saponified stock thus obtained to distillation in an intermittent or batch distillation system, with the aid of superheated steam injection into the charge, the entire system being maintained under diminished pressure. The stills employed in such distillation are generally heated by direct fire, in which case the bottom of the still is always at a considerably higher temperature than the temperature of the major portion of the material contained therein. That is, there are local areas of highly heated stock adjacent the metal of the still. Invariably, this high temperature, and the extended period of heating, causes a thermal decomposition of constituents of the stock, giving rise to a viscous residue which cannot be further purified without great cost. Also, the distillate is of poor color and odor. In some instances superheated steam and high vacuum have been employed in distilling the stock, but this method has proved unsatisfactory from many standpoints, such as cost, time and efficiency of recovery of the fatty acids.

A primary object of the present invention is the provision of a process for distilling fatty acid materials in an improved manner.

An additional object is to provide a process for distilling fatty acids in which material decomposition is avoided.

A further object is the provision of an improved process for treating raw fatty acid stock containing free fatty acids and neutral oil.

A still further object is the provision of a process for separating the various constituents of a mixture of different fatty acids.

These and other objects will be apparent from a consideration of the following illustrative and explanative description and with reference to the accompanying drawing, in which the single figure represents, diagrammatically, suitable apparatus in which to carry out our improved process.

In accordance with our invention, the stock, containing free fatty acids, is heated quickly to a temperature of incipient vaporization, and then subjected to flash distillation to remove the fatty acids therefrom. The neutral oil remaining as an undistilled residue may be subjected to further treatment, such as saponification, while the distillate, which contains a mixture of fatty acids, may be subjected to further treatment, such as fractionation, whereby to separate the various acids.

The apparatus shown in the drawing is somewhat similar, in many respects, to that employed, for example, in the petroleum industry.

The charge of stock is run into two deodorizers 10 and 11 wherein volatile color and odor imparting impurities and moisture are removed by means of closed steam coils 12 and 13 and open steam coils 14 and 15, the impurities and moisture passing from the deodorizers through pipe 16 into a suitable condenser 17. The deodorizers are preferably maintained under a partial vacuum by means of a suitable vacuum line 18 connected to the condenser 17. When a sufficient charge of stock is admitted to the deodorizers, steam is turned into the closed coils 12 and 13 and steam is passed directly into the charge by means of the open coils 14 and 15. Under the elevated temperature and steam injection, volatile impurities and moisture are removed within a few hours and the fatty acid material is ready for subsequent operations. It will be understood that the deodorizers 10 and 11 are used intermittently, that is, while the stock in one deodorizer is being treated, the other deodorizer may be used

as a source of stock for the subsequent treatments. In this manner the operation of the remainder of the apparatus is made continuous. Primarily, the deodorization serves to remove certain light unsaturated products, volatile color and odor imparting bodies, permanent gases, and moisture from the stock, the presence of these impurities being objectionable in the subsequent treatment to which the stock is subjected, as well as in the final products. For instance, moisture vaporizes in the pipe still, to be described hereinafter, with explosive violence, thereby causing undue entrainment and subsequent contamination of the distillate.

When sufficiently deodorized, the stock is transferred through line 19 by means of pump 20 into a pipe still 21. The still is provided with a fire box 22 and a steam superheater 23, the latter being used to supply steam for the various open coils of the apparatus. A meter 24 is connected in line 19 by means of which the rate of flow of stock through the line is indicated. As the stock passes through tubes 25 of the pipe still, which define a tortuous passageway, they are raised quickly to a temperature which is high enough to impart a sufficient quantity of sensible heat to the stock above that required for the vaporization of the fatty acids at reduced pressure to cause their distillation when subsequently passed into a flash chamber maintained at reduced pressure.

In the batch stills of the prior art the fatty acids remain in the high temperature of the still about eight or ten hours, the stills being maintained at about a 10,000 lb. charge, distilling at the rate of about 1,000 lbs. per hour. Also, as before stated, such stills have local areas which are heated considerably higher than the temperature of the mass, the time involved, and the high temperature causing objectionable deterioration of the materials. Our process is so regulated that the stock passes through the pipe still in a few minutes, the large area of heating surface being sufficient to cause a very rapid increase in the temperature of the stock.

Experiments show that fatty acid stock is subject to thermal decomposition, which proceeds more rapidly as the material is continuously vaporized. In accordance with our invention, the stock is controlled while in the pipe still so that the vaporization therein is maintained at a minimum. This may be accomplished by suitable control of the pressure within the still, a higher pressure acting to retard vaporization. When the heated stock enters the flash chamber, vaporization is practically instantaneous and decomposition is thereby reduced to a minimum.

The stock preferably is heated in the deodorizers by the open and closed steam coils therein to a temperature of the order of 300° F., and are transferred to the pipe still at substantially the same temperature. By controlling the temperature of the fire box 22, the length and size of pipes in the pipe still, and the velocity of the material transferred therethrough, it is possible to obtain any desired result with respect to temperature and time of treatment. In a preferred modification, the speed of the stock through the pipe still is on the order of two feet per second, substantially six minutes being required for the stock to pass completely through the still. The temperature of the still is preferably controlled so as to raise the temperature of the stock to substantially 450° to 550° F., a slight pressure, sufficient to retard vaporization, being maintained within the pipe still; the exact temperature and pressure being dependent upon such factors as the nature of the particular material being treated.

A steam line 26 communicates with the pipe still at a point intermediate the length of the pipe 25 thereof, whereby steam, preferably superheated, is added to the stock during the heating process, creating a partial pressure that will lower the boiling point and assist in the subsequent vaporization to maintain a low distillation temperature.

It will be seen that the heating period of our process, which may be from 4 to 10 minutes, for example, compares very favorably to the heating period of 8 to 10 hours which is employed in batch stills. The heat is applied evenly by the pipe stills, and in the short time involved, thermal decomposition is reduced to the minimum.

Although, as described heretofore, it is preferable to retard vaporization in the pipe still, in some cases it may be found desirable to allow incipient vaporization in the pipe still, and to complete the vaporization by subsequent introduction into a flash chamber. In this case, it is not necessary to heat the stock to as high a temperature as where sufficient sensible heat must be added to the liquid stock to substantially completely vaporize the volatile constituents thereof. It will be understood that the particular pressure in the flash zone will depend upon such factors as the type of equipment employed, the nature of the material treated, and the rate of distillation. By way of example, it may be stated that a pressure of 45 to 60 mm. Hg. has been employed to advantage in the flash zone. In such instances, the pressure in the upper part of the fractionating tower through which the vapors subsequently are passed may be of the order of 10 mm. Hg.

The heated stock passes from the pipe still into a valve controlled line 27 which communicates with the interior of a flash chamber 28. The stock, heated to a temperature which may be between 450° and 550° F., is released into the flash chamber which is maintained under reduced pressure. It is preferred to spray the stock into the flash chamber to facilitate subdivision and vaporization of the fatty acids therein. The sensible heat in the stock is sufficient to cause a flash distillation of the volatile constituents thereof, that is, to supply the latent heat of vaporization. The temperature of the distillation of the stock will ordinarily be in the neighborhood of 400° F., which is considerably below the temperature of the stock in the pipe still, thereby preventing excess decomposition which would result from vaporization of the stock at a high temperature.

In the case of said stock being introduced into the flash chamber the fatty acid content is vaporized, while the remainder of the stock, which is mostly neutral oil, retains its liquid form.

The flash chamber is preferably a vessel of fairly large volume and may contain a number of bubble trays. Above the point at which the stock is introduced into the flash chamber are mounted a series of mist extractors 29 which serve to remove entrained unvaporized materials from the fatty acid vapors. The entrained liquid, together with the unvaporized portions of the stock, falls toward the bottom of the flash chamber and is received by a series of bubble trays 30 of suitable construction. As the unvaporized residue of the stock flows downwardly over the trays 30 it is met with a rising current

of steam from the open coil 31 positioned in the lower portion of the flash chamber, thereby stripping this unvaporized residue of its residual portion of fatty acids. This steam and the removed residual portions of the fatty acids pass upwardly through the flash chamber and are mingled with the fatty acids volatilized during the distillation process.

Communicating with the bottom of the flash chamber is a tank 32 in which the unvaporized residuum of the stock is collected. This residuum may be collected until a sufficient quantity is obtained for further treatment. In the case of raw stock, the portion which is not vaporized contains quite a large percentage of neutral oil. Since the raw stock already contains a considerable quantity of free fatty acids, the further splitting or saponification was expensive and inefficient, a greater amount of handling being required due to the large volume of material and a larger quantity of reagents being used to provide the proper concentration. By first distilling the free fatty acids from the raw stock by the method described herein and then splitting the neutral oil in the residue by the Twitchell process or other treatment, a higher percentage of recovery of fatty acid and glycerine is obtained. After splitting the residue, as by the Twitchell method, it is possible to recover the glycerine from the mixture, the fatty acids being again distilled as described heretofore. The distillation may be effected by adding the split material to a fresh batch of stock. Heretofore millions of pounds of glycerine have been lost by the costs necessary to recover the same from raw stock. When recovered in accordance with our process this glycerine is of a good grade and the process economical.

In other words, where the raw material, which may contain on the order of 60 per cent free fatty acids, is subjected to a further splitting in accordance with Twitchell's process, the entire amount of the oil is treated by boiling for 18 hours in admixture with about 2 per cent reagent, 1 per cent sulfuric acid, and 40 per cent of water by weight of the whole batch of oil, including the 60 per cent free fatty acids. A second boil is generally made with substantially 0.5 per cent acid and 10 per cent additional water. The total split from this operation is about 94 per cent. There results from this operation a glycerine water of about 2 per cent or 3 per cent concentration. In accordance with my invention, the raw material is first distilled as described to remove the free fatty acids, and then subjected to the splitting operation, whereby only about 40 per cent of the material is handled and a glycerine water of about 8 per cent concentration is obtained. Glycerine of good quality may be obtained from this water at a reasonable cost.

Operation of our process will depend in detail upon the particular type of fatty material employed and the content of fatty acids.

Where saponified stock or the fatty acid material resulting from the splitting of the residue of the distillation of raw stock is redistilled without being mixed with fresh stock, the residue obtained is preferably converted directly into stearine pitch.

Operations have shown that by resplitting the material as described heretofore there is an increase in yield of fatty acids between 4 per cent and 8 per cent.

Heretofore considerable efforts have been made to separate the various constituents of a mixture of distilled fatty acids obtained from splitting a neutral fat or oil, such as cottonseed oil or tallow. Where the stock distilled has been obtained from cottonseed oil, the composition of the distilled free fatty acids may be approximately 50 per cent linoleic acid, 25 per cent oleic acid, and 25 per cent palmitic acid. The oleic and the linoleic acids are unsaturated acids which are liquid at ordinary temperatures. Oleic acid congeals at about 4° C. and linoleic acid at about —16° C. Palmitic acid, on the other hand, is a saturated solid acid having a melting point of 62° C. These acids have been used in the soap industry for many years. There has been a demand for higher titered fat, such as palmitic acid, for use in the composition of many soaps, it being recognized that a saturated fat is more desirable in this connection. Considerable effort has been made toward the separation of the palmitic constituents of the fatty acid mixture in the production of soaps, one method which has gone into extensive use consisting in subjecting the mixture of fatty acids to a fractional crystallization. However, the method has proved unsatisfactory, due to the inefficient degree of removal, the high cost and the cumbersome apparatus necessary to carry out the process.

In accordance with our invention, the constituents of the fatty acid mixture are separated by a fractional distillation process in which the fatty acids vaporized in the flash chamber pass upwardly through the mist extractors 29, through a line 33 communicating with the top of the flash chamber and into a fractionating tower 34 at an intermediate portion thereof. In the lower portion of the fractionating column or tower 34 are a series of bubble plates 35 of suitable structure and design. The fatty acid vapors entering the fractionating tower arise therethrough and are met with a downwardly flowing stream of a reflux which is preferably composed of the condensate of the lighter fraction of the fatty acids which is taken from the top of the fractionating column. This condensate flows downwardly across a second series of bubble plates 36 similar in design to the plates 35. In traveling upwardly through the fractionating column the higher boiling constituents of the fatty acid vapors are condensed by the reflux and the material on each succeeding higher plate is of a lower boiling point. That is, the fractionating column reaches the state of equilibrium in which the composition of the material on each plate remains substantially constant and the boiling point of the material on each higher plate is lower than the liquid on the next lower plate.

Somewhere in the fractionating column will be found a plate which will contain the largest percentage of linoleic acid. The liquid is drawn from this plate by line 37 and passed into a stripper 38. In the bottom of this stripper is an open steam coil 39, the live steam passing through the liquid acting to remove residual portions of palmitic acid, the vapors of palmitic acid returning directly to the column through line 39. A line 40 communicates with the bottom of the stripper and passes, through a suitable pump, to a storage tank. This liquid is fairly pure linoleic acid but will be found to contain a certain amount of oleic acid, a more complete separation, if desired, being dependent upon further treatment. The stripper contains an open steam coil 41 in its bottom portion, by means

of which the lower boiling palmitic acid is stripped from the linoleic acid mixture.

The liquid collecting in the bottom of the fractionating column, consisting primarily of oleic acid, is drawn off through line 42 and passed through a pump to a suitable place of storage. Fractionation in the lower portion of the fractionating tower is made possible by passing a current of steam from the open steam coil 43 positioned in the bottom of the fractionating column upwardly through the succeeding bubble plates, the downwardly flowing fatty acid mixture acting as reflux and being separated into portions of successively higher boiling point toward the bottom of the tower.

From the upper bubble plate 36 of the fractionating column the vapors, consisting principally of steam and palmitic acid, pass through a partial or reflux condenser 43 which furnishes sufficient reflux for the desired fractionation. From the reflux condenser 43 the vapors, which consist primarily of steam and palmitic acids, pass through a line 44 into a vapor condenser 45. The condenser preferably is provided with large vapor spaces, thereby permitting a minimum pressure drop across it. The temperature of the condenser is so regulated that practically all of the palmitic acid vapor is liquefied, the condensate being drawn off through a line 46 and transferred through a pump to any suitable source of disposition.

The vapors which leave the condenser 45 will preferably be almost entirely steam. However, experience has shown that the steam will contain an appreciable amount of mechanically incorporated or entrained fatty acids in the form of mist. To remove the entrained fatty acids from the steam, the steam vapors are passed through a mist remover or catch-all 47, the temperature of which is preferably above the melting point of the fatty acids, whereby the particles of fatty acid are removed.

The fatty acid passing from the fractionating column and liquefied in the condenser 45 is substantially pure palmitic acid. It will be seen from the foregoing description that we have provided a process by which animal or vegetable fatty acid mixtures may be separated into their various constituents, thereby making the latter available for many uses to which the mixture of fatty acids are not adaptable.

In some instances it may be found desirable to flash the heated stock directly from the pipe still into the fractionating column, rather than into a separate flash chamber as described hereinbefore. This is particularly true where it is desired to operate at as low a pressure as possible.

The steam after being scrubbed of its suspended fatty acid mist is passed directly to a barometric condenser 48 to which is connected suitable vacuum pumps, not shown. These pumps serve to maintain the entire system, including the flash chamber and fractionating tower, under a suitable reduced pressure.

It will be observed by one skilled in the art that the foregoing specific description is subject to modification, and such variations as come within the spirit of our invention are intended to be included in the appended claims.

We claim:

1. The process of treating oleaginous materials containing a mixture of volatile fatty acids having different boiling points, which comprises passing said material through a tortuous passage subject to a temperature sufficiently high to raise the temperature of the material higher than necessary for vaporization of fatty acids at reduced pressure and sufficiently high for flash distillation, maintaining said materials under pressure during the heating period, subjecting said materials to flash distillation in a zone of reduced pressure and forming a mixture of vapors containing said fatty acids heated sufficiently high for fractional distillation, and subjecting said vapors to a fractional distillation procedure for the separation of the fatty acids into volatile fractions having different boiling points, said procedure including the steps of passing the heated vapors through a series of heat-exchanging pools of condensed fatty acid mixtures having successively lower boiling points, condensing the low-boiling vapors passing through the last of said pools, returning a portion of said condensed low-boiling vapors to said pools as reflux liquid, and withdrawing from said pools a liquid volatile fatty acid fraction of higher boiling point than said low-boiling fraction.

2. The process of treating oleaginous materials containing a mixture of volatile fatty acids having different boiling points, which comprises subjecting said materials to continuous distillation in which said fatty acids are heated quickly to a temperature above the temperature required for vaporization, at reduced pressure, then producing under reduced pressure a mixture of vapors containing said fatty acids and being heated sufficiently higher than that temperature which is the boiling point of said vapors at the pressure thereof, for fractional distillation, and subjecting said heated vapors to a fractional distillation procedure for the separation of the fatty acids into volatile fractions having different boiling points, said procedure including the steps of passing the heated vapors through a series of heat-exchanging pools of condensed fatty acid mixtures having successively lower boiling points, condensing the low-boiling vapors passing through the last of said pools, returning a portion of said condensed low-boiling vapors to said pools as reflux liquid, and withdrawing from said pools a liquid volatile fatty acid fraction of higher boiling point than said low-boiling fraction.

3. The process of treating oleaginous materials containing a mixture of fatty acids having different boiling points, which comprises subjecting said materials to a continuous distillation step in which said fatty acids are heated quickly to a temperature of incipient vaporization above the temperature required for vaporization at reduced pressure and producing a mixture of vapors at reduced pressure containing said fatty acids heated sufficiently for fractional distillation, and subjecting the heated vapors to a fractional distillation procedure for the separation of the fatty acid into volatile fractions having different boiling points, said procedure including the steps of passing the heated vapors through a series of heat-exchanging pools of condensed fatty acid mixtures having successively lower boiling points, condensing the low-boiling vapors passing through the last of said pools, recirculating a major portion of said condensed low-boiling vapors to said pools as reflux liquid for reboiling at a lower temperature than the temperature of said heated vapors and withdrawing from the lower of said pools a liquid volatile fatty acid fraction of higher boiling point than said low-boiling fraction, said higher boiling fraction being heated to a temperature above the temperature of the upper of said pools.

RALPH H. POTTS.
JOHN E. McKEE.