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APPARATUS FOR THE DISTILLATION OF LIQUIDS HAVING RELATIVELY HIGH BOILING POINTS

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Fig. 1.

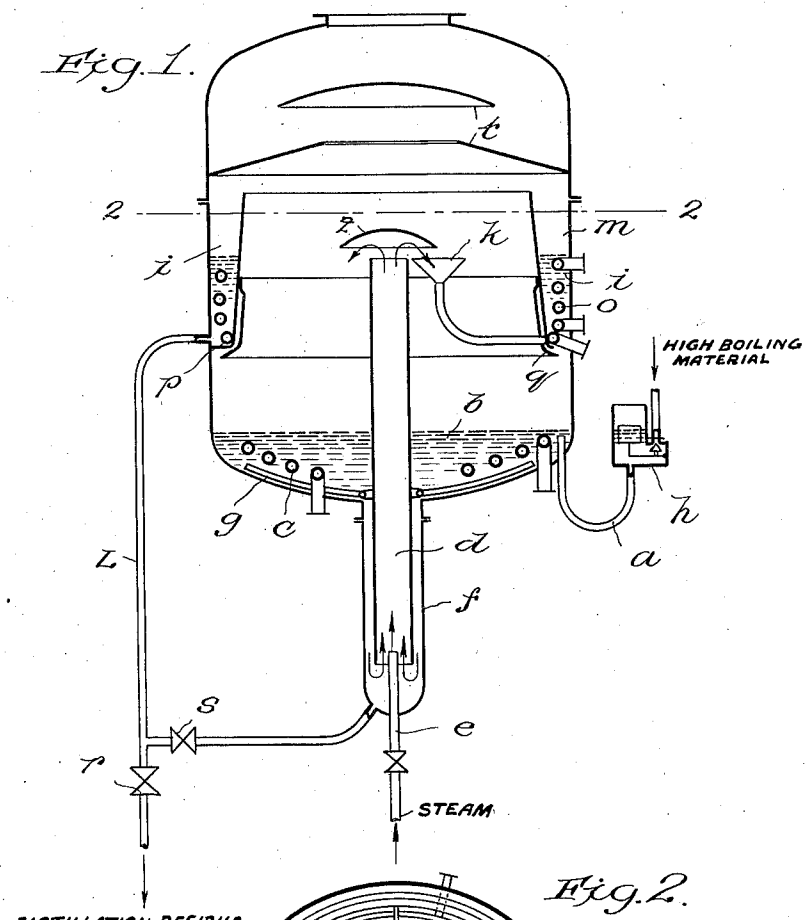
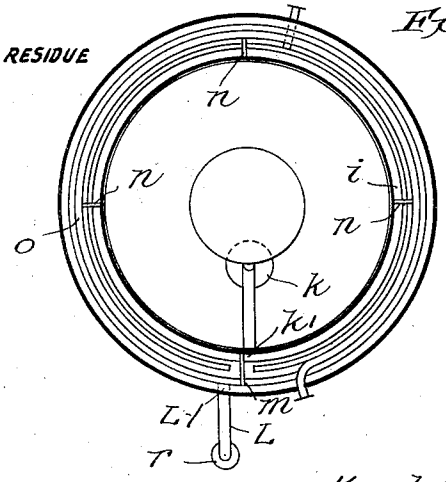


Fig. 2.



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APPARATUS FOR THE DISTILLATION OF LIQUIDS HAVING RELATIVELY HIGH BOILING POINTS

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10 Claims. (Cl. 202—175)

The present invention relates to the distillation of liquids having relatively high boiling points, and, more particularly, to an apparatus for the continuous, stage distillation of liquids such as fatty acids, glycerin, and the like.

Heretofore, the conventional processes for the continuous distillation of liquids having high boiling points, such as fatty acids, glycerin, and the like, have been carried out in such a manner that the liquid to be treated was conducted at a low rate of flow through an evaporator. At the same time, the quantity of liquid introduced into the apparatus has been so controlled that by the time the liquid passed through the apparatus, the distillable components were evaporated to the desired extent. Various types of apparatus were employed for carrying out these conventional processes. One type of conventional apparatus involved horizontal troughs provided with vertical separating walls through which the liquid to be distilled was caused to stream in horizontal direction. Other types of conventional apparatus provided a vertical flow of the liquid and have been provided with distributing nozzles and discs. Considerable difficulty was experienced in the design of apparatus of the latter type which required special means for conducting the liquid to be distilled in contact with the heating surfaces. Another disadvantage of these processes was that the movement of the liquid along the heating surfaces was very little so that larger heating surfaces have been required for the indirect heating. Moreover, the slow streaming of the liquid through the apparatus greatly reduced the speed of the heat exchange within the liquid, frequently causing localized overheating and decomposition of the liquid, which not only greatly reduced the yield of distilled product but at the same time also detrimentally influenced the quality of the final product. It has also been suggested to employ distillation columns provided with a suitable plurality of channel-like distillation spaces arranged one above the other, said channels having a high outer edge and a low inner edge. The liquid subjected to distillation was introduced into the channels and was caused by steam agitation to pass over the low inner edges of the channels to the lower channels through the intermediary of drip plates and similar devices. In each of the channels, heating means have been arranged close to the bottom thereof but outside of the liquid space. Somewhat better results have been obtained by another conventional evaporator in which steam has been directly injected into the liquid to be distilled whereby substantial quantities of the liquid were propelled upwards and were allowed to descend again along suitable surfaces. This type of apparatus and process provided very lively circulation of the liquid and reduced the danger of local overheating but had serious disadvantages when carrying out the process continuously. Since the circulated quantity of the liquid was multiple of that of the distilled quantity, the content of the distillation chamber had a composition similar to that of the residues to be discharged. These residues generally were very viscous and greatly reduced the intensity of the movement or agitation of the liquid. A further disadvantage experienced in this type of process was the relatively high operating temperatures since also the fractions of a higher boiling point had to be distilled off. Although various other suggestions and proposals have been made, none, as far as is known, was completely satisfactory and successful when carried into practice on a commercial and industrial scale.

It has been discovered that the outstanding problem can be solved in a remarkably simple manner.

It is an object of the present invention to provide an improved process for the distillation of liquids having relatively high boiling points, which is free from the above disadvantages and inconveniences of conventional distillation processes.

It is another object of the present invention to provide a novel and improved process for distilling liquids having high boiling point, such as fatty acids, glycerin, and the like, which involves distilling the liquids in two steps or stages.

The invention also contemplates a distillation process for liquids of the character described comprising a preliminary distillation stage after which the liquid is transferred by means of steam agitation to a secondary or auxiliary distillation stage where the distillation is finished and the residue is withdrawn

It is also within contemplation of the present invention to provide a novel apparatus for carrying the process of the present invention into practice on a practical and industrial scale.

Other and further objects and advantages of the invention will become apparent from the following description taken in conjunction with the accompanying drawing in which—

Fig. 1 illustrates a vertical sectional view of a distillation apparatus embodying the principles of the present invention; and

Fig. 2 depicts a cross section taken on line 2—2 of Fig. 1.

Broadly stated, the present invention permits obtaining the combined advantages of all of the conventional processes without any of the disadvantages thereof. According to the principles of the present invention, the distillation is carried out in two steps or stages in a distillation vessel or still. The fatty acids, glycerin or other similar liquids having relatively high boiling points are introduced into the lower portion of said vessel to form a body, pool or bath of liquid therein. This body of liquid is subjected to the effect of direct and indirect heating and to powerful vertical circulation sufficient to carry a substantial portion of the liquid above the level of said body of liquid. A partion of the vertically circulated liquid is retained or trapped and is introduced into a secondary or auxiliary distillation space or chamber forming a secondary body of liquid therein. In this secondary distillation chamber, the distillation is finished and the distillation residues of the desired composition are withdrawn. In this manner, the advantages of powerful circulation are assured for the greatest portion of the produced distillates, while the disadvantages otherwise connected with the continuous operation have no effect on the preponderant quantity of the distillate produced from the final or auxiliary distillation stage.

The invention will now be more fully explained to those skilled in the art in connection with the accompanying drawing. Referring particularly to Figs. 1 and 2 of the drawing, the liquid to be distilled is introduced through a conduit *a* into a lower distillation chamber *b* of the apparatus. The lower distillation chamber is provided with a heating coil *c* located at the bottom thereof for introducing the heat required for effecting the distillation. The circulation and the continuous agitation of the liquid to be distilled along the heating coil *c* is accomplished by means of a centrally arranged circulation tube *d* into which steam is introduced through conduit *e*. The bottom of the distillation apparatus is connected with a dip pipe *f* which surrounds the lower portion of the circulation pipe *d*. The circulation pipe is of such length that an effective and powerful circulation of the liquid is obtained. By means of the described arrangement of the heating coil *c* to which a heating medium for instance steam is introduced by suitable means preferably through the bottom of the apparatus and of the dip pipe *f*, it is possible to reduce the lower liquid or distillation space to very small dimensions.

The direct steam necessary for the distillation and for improving the powerful agitation of the liquid along the heating coil is introduced and uniformly distributed by means of a nozzle pipe *g* arranged at the bottom of the distillation space and connected to a steam line. Preferably, an automatically actuated inlet valve *h* is provided in the conduit or pipe *a* introducing the liquid to be treated, which admits only so much liquid as has been removed by distillation so that the liquid is maintained at a constant level in the lower distillation space or chamber.

The distillation in the lower distillation space is carried to a certain degree of concentration. In other words, it is carried on until a definite and preferred proportion between the distillable and undistillable constitutents of the liquid is obtained.

During the course of the distillation process, a portion of the circulated liquid is continuously removed and is transferred into the upper secondary or auxiliary distillation space *i* of annular cross section. This is accomplished by means of a funnel or some other equivalent member *k* which traps a portion of the liquid circulated by the circulation pipe *d* and deflected by a deflector plate *z* and through a suitable conduit which conveys the trapped liquid into the upper or auxiliary distillation space or chamber. The level of the liquid in the upper distillation chamber *i* is determined by the position of the upper edge of funnel *k*. In this upper distillation space *i* the final distillation is conducted of the distillable portion of the liquid which is thus separated from the undistillable residue. The distillation residue is continuously discharged through a conduit L. The quantity of liquid residue removed through conduit L automatically controls the quantity of fresh liquid within distillation space *i* so that it is unnecessary to provide special controlling means to this purpose.

During the distillation in the upper distillation chamber *i*, the liquid is positively conducted around the annular distillation space. This is accomplished by means of a vertical separating wall *m* mounted in the annular distillation chamber which causes the liquid introduced by funnel *k* and entering the distillation space at *k*—*l* to flow completely around space *i* up to the mouth L—*l* of conduit L through which the distillation residue is discharged. The path of the liquid from *k*—*l* to L—*l* is of such length and the heating effect of coil *o* is so controlled that sufficient time is provided for the distillation during the period the liquid arrives from *k*—*l* to L—*l*. Preferably, vertically arranged baffle plates *n* are provided along the path of the streaming liquid from *k*—*l* to L—*l*, in order to prevent mixing of the continuously moving liquid entering at *m* with the liquid leaving the distillation chamber at L.

The upper distillation chamber is indirectly heated preferably by steam which is introduced into a heating coil *o*. A circular nozzle pipe *p* introduces direct steam required to the distillation and uniformly distributes the steam along the full circumference of the upper distillation chamber *i*. The operating pressure of the heating steam in the upper heating coil *o* is higher than that of the lower coil *c* because the distillation in the upper distillation chamber requires higher temperatures. For this reason, it is advisable to provide the upper distillation chamber with a double wall *q* which prevents excessive radiation of heat and overheating of the liquid propelled upwards from the lower distillation chamber *b*.

The apparatus embodying the principles of the invention may be also used for semi-continuous operation or for operation by individual charges. This can be accomplished by throttling or completely closing control valve *r* in conduit L and by opening closure valve *s* whereby continuous circulation is obtained between the upper distillation chamber *i* and the lower distillation chamber *b* through the intermediary of circulation pipe *d* and funnel *k*.

In the upper portion of the apparatus, a separator *t* is provided which separates the liquid particles carried over by the distilled vapors from the upper and lower distillation chambers. The separated particles of the liquid may be returned, for example, to the lower distillation chamber *b*.

It is to be noted that the invention provides a

number of important advantages. First of all, the quantity of liquid present in the apparatus is very small so that the undistillable part of the liquid is only exposed for a relatively short time to the effect of the distillation temperatures whereby losses and deterioration of the liquid by decomposition are prevented.

It is also to be observed that the liquid is maintained in lively agitation and movement along the heating surface which causes very favorable heat exchange conditions so that only low temperature differences are required between the heating surface and the liquid to be distilled and in addition, the distillation can be carried out at relatively low temperatures.

Moreover, local overheating of the liquid is effectively prevented and relatively high yields are obtained with an apparatus of simple and inexpensive construction and of relatively small dimensions.

Although the present invention has been described in connection with a preferred embodiment thereof, variations and modifications may be resorted to by those skilled in the art without departing from the principles of the present invention. All of these variations and modifications are considered to be within the true spirit and scope of the present invention as disclosed in the present description and defined by the appended claims.

The present application is a division of my copending application Ser. No. 100,117, filed on Sept. 10, 1936, now Patent 2,179,833 and having the title "Distillation of liquids having relatively high boiling points."

I claim:

1. In an apparatus for distilling liquids having relatively high boiling points, the combination comprising a substantially cylindrical distillation vessel having a bottom portion adapted to form a preliminary distillation chamber, a centrally located dip pipe communicating with said chamber, direct and indirect steam supplying means in said chamber for heating a liquid therein and for agitating said liquid, an annular auxiliary distillation chamber associated with an intermediate portion of said vessel, a circulation pipe mounted in said dip pipe and extending into said vessel, means for passing steam through said circulation tube to cause vertical circulation of said liquid, means for trapping a portion of said vertically circulated liquid and for transferring said trapped portion into said auxiliary distillation chamber to finish the distillation therein, means for withdrawing distillation residues from said auxiliary distillation chamber, and a vertical partition wall in said auxiliary distillation chamber causing said liquid to flow around in said chamber from the point of introduction to the point of discharge.

2. In a distillation apparatus for distilling liquids having relatively high boiling points, the combination comprising a distillation vessel, a lower distillation chamber associated with the bottom portion of said vessel, an annular upper distillation chamber associated with an intermediate portion of said vessel, direct and indirect steam supplying means for said chambers to heat liquid therein and to agitate said liquid, means including a steam actuated vertical circulation pipe for transferring partially distilled liquid from said lower to said upper chamber, and a vertical partition wall in said upper chamber causing said transferred liquid to flow around from its point of admission to its point of discharge.

3. In an apparatus for distilling liquids having relatively high boiling points, the combination comprising a distillation vessel, a distillation chamber therein adapted to hold a body of liquid to be distilled and associated with the lower portion of said vessel, an annular distillation chamber having its vapor space in common with the first distillation chamber, means for heating the liquid in said first chamber, means for admitting steam to agitate and vertically circulate said liquid and to raise said liquid above the level of said annular chamber, means for trapping a portion of said vertically circulated liquid and for transferring the same into said annular distillation chamber, and heating means associated with said annular chamber to continue distillation of the liquid therein.

4. In an apparatus for distilling liquids having relatively high boiling points, the combination comprising a distillation vessel, a dip pipe communicating with the bottom portion of said vessel forming a first distillation chamber adapted to hold a liquid to be distilled, means for heating and agitating said liquid, a circulation tube mounted in said dip pipe and extending upward into an intermediate portion of said vessel, an annular distillation chamber associated with said intermediate portion of said vessel and having a common vapor space with said first distillation chamber, means for passing a vapor through said circulation tube to agitate said liquid, to cause vertical circulation of said liquid, and to raise a portion of said liquid above the level of said annular chamber, means for transferring a portion of said vertically circulated liquid into said annular distillation chamber, means for heating said annular chamber to continue distillation of the liquid therein, and means for withdrawing distillation vapors from said vessel.

5. In an apparatus for distilling liquids having relatively high boiling points, the combination comprising substantially the apparatus described in claim 4 together with means for withdrawing distillation residues from the annular distillation chamber therein described, and a vertical, radially placed partition wall in said annular chamber causing the liquid therein to flow around in said chamber from the point of introduction to the point of discharge.

6. In an apparatus for distilling liquids having relatively high boiling points, the combination comprising a distillation vessel, a distillation chamber within said vessel, an annular distillation chamber within said vessel having a vapor space in common with the first distillation chamber, a radial partition wall in said annular distillation chamber, means located in both distillation chambers for heating the liquid by means of a fluid medium, conduit means for transferring liquid from said first distillation chamber into said annular distillation chamber and ending at one side of said partition wall, an outlet for distillation residues at the other side of said partition, and means for withdrawing distillation vapors from the common vapor space.

7. In an apparatus for distilling liquids having relatively high boiling points, the combination comprising a distillation vessel, a distillation chamber within said vessel, an annular distillation chamber within said vessel having a vapor space in common with the first distillation chamber, a radial partition wall in said annular distillation chamber, means located in both distillation chambers for heating the liquid by means of a fluid medium, means located in both distilla-

tion chambers for introducing steam into the liquid, conduit means for transferring liquid from said first distillation chamber into said annular distillation chamber and ending at one side of said partition wall, an outlet for distillation residues at the other side of said partition, and means for withdrawing distillation vapors from said common vapor space.

8. In an apparatus for distilling liquids having relatively high boiling points, the combination which comprises a distillation vessel having a lower portion adapted to form a distillation chamber, an annular distillation chamber within said vessel and having a vapor space in common with the first distillation chamber, means in said annular distillation chamber constructed and arranged to provide horizontal flow of the contents of said annular distillation chamber, means for conveying portions of a liquid in the first distillation chamber to a point above the level of said annular chamber, means cooperating with said liquid-raising means to conduct a portion of the liquid thus raised into said annular distillation chamber, means for introducing liquid to be distilled into said first distillation chamber, means for heating the contents of said first distillation chamber and said annular distillation chamber, and means for removing liquid from said annular distillation chamber.

9. In a distillation apparatus for distilling liquids having constituents with relatively high boiling points for separating distillable constituents from undistillable constituents, the combination which comprises a distillation vessel having a substantially closed top, a vapor outlet provided in said top for removing distilled vapors, a distillation chamber associated with the lower portion of said vessel adapted to hold a body of liquid to be distilled, means for heating liquid in said chamber to distil distillable constituents and to obtain a definite proportion between distillable constituents and undistillable constituents, and an auxiliary distillation chamber adapted to hold a body of liquid to be distilled and arranged to maintain a secondary body of liquid separate from the first body of liquid in the first distillation chamber, means for heating the liquid in the auxiliary distillation chamber for effecting distillation of a distillable portion from an undistillable portion, means for circulating the liquid in the first distillation chamber and by-passing a portion of such circulated liquid into the auxiliary distillation chamber, a vapor chamber associated with said vapor outlet and common to both distillation chambers, and means associated with said auxiliary distillation chamber for removing an undistilled portion whereby liquids having constituents with relatively high boiling points are separated into distilled portion and undistilled portion while subjecting the undistillable portion of the liquid to the effect of distillation only for a relatively short time thereby preventing losses and deterioration of the liquid by decomposition.

10. In a distillation apparatus for distilling liquids having constituents with relatively high boiling points for separating distillable constituents from undistillable constituents, the combination which comprises a distillation vessel having a substantially closed top, a vapor outlet provided in said top for removing distilled vapors, a distillation chamber associated with the lower portion of said vessel adapted to hold a body of liquid to be distilled, means for heating the liquid in said chamber to distill distillable constituents and to obtain a definite proportion between distillable constituents and undistillable constituents, an annular auxiliary distillation chamber associated with the first distillation chamber and adapted to hold a body of liquid to be distilled and arranged to maintain a secondary body of liquid separate from the first body of liquid in the first distillation chamber, means for heating the liquid in the annular auxiliary distillation chamber for effecting distillation of a distillable portion from an undistillable portion, means for circulating the liquid in the first distillation chamber and by-passing a portion of such circulated liquid into the annular auxiliary distillation chamber, a vapor chamber associated with said vapor outlet and common to both distillation chambers, and means associated with said annular auxiliary distillation chamber for removing undistilled portion of said liquid whereby liquids having constituents with relatively high boiling points are separated into distilled portion and undistilled portion while subjecting the undistillable portion of the liquid to the effect of distillation only for a relatively short time thereby preventing losses and deterioration of the liquid by decomposition.

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