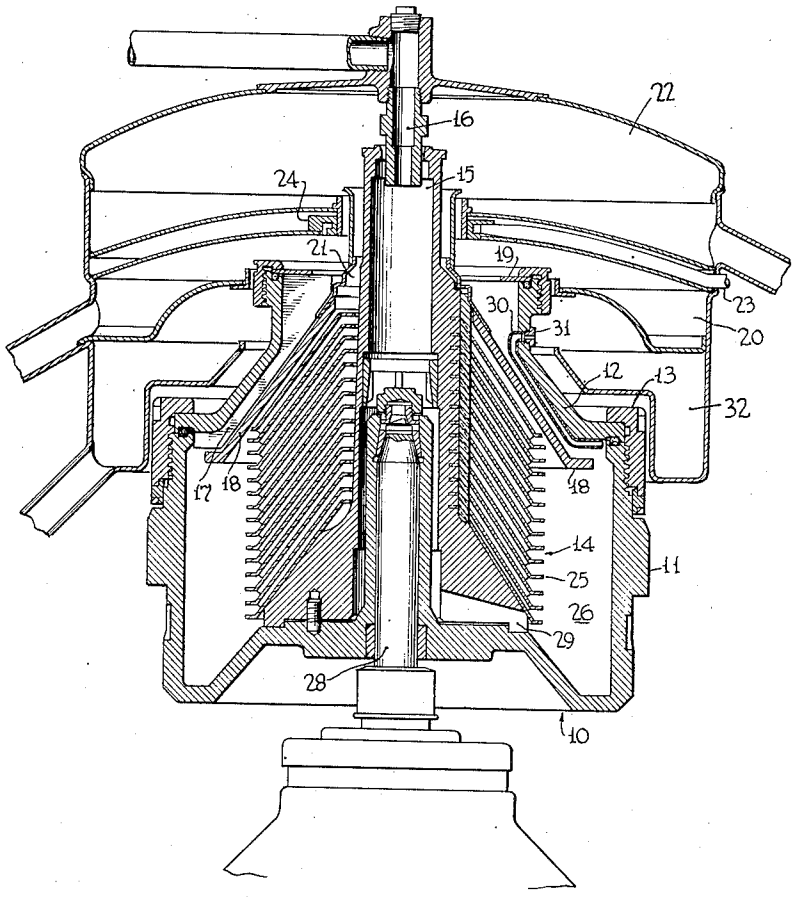
Dec. 16, 1941. L. D. JONES 2,266,554

PROCESS OF DEWAXING HYDROCARBON OILS

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PROCESS OF DEWAXING HYDROCARBON OILS

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9 Claims. (Cl. 196—19)

The present application is a continuation in part of my prior application, Serial #243,559, filed on December 2, 1938, for Process and apparatus of dewaxing hydrocarbon oils.

The present invention pertains to the art of separating mixtures of liquids and solids by subjecting such mixtures to the influence of centrifugal force to effect subsidence thereof in concentric layers and discharging the materials of these layers separately from the influence of centrifugal force. It is concerned primarily with a process of effecting such separation and separate discharge, but includes as an additional feature a machine having particular utility for performing the steps constituting the process of the invention. While both the process and machine of the invention will probably be found useful in the solution of various separation problems, the invention was conceived as a solution of problems arising incident to the separation of wax from petroleum stock, and has been found to be particularly useful in connection with these particular problems. In the following description, the invention will therefore be discussed in its relationship to separation of wax from oil, it being understood that this method of description is adopted merely for the purposes of convenience of illustration, and not with any intent to limit its scope to this single use.

The invention may be considered as an improvement over the process of dewaxing petroleum stocks described in the patent to D. T. Sharples No. 1,351,265 of August 31, 1920. That process has gone into extensive commercial use in the United States and foreign countries. In the practice of the process of that patent, the stock to be dewaxed is first diluted with a sufficient amount of relatively low-boiling hydrocarbon solvent to render the oil phase, upon subsequent chilling to precipitate wax, distinctly lighter than the precipitated wax. The diluted stock, containing both oil and wax, is thereafter chilled to a temperature below 0° F. in order to precipitate the wax from solution in the diluent. The oil solution containing the precipitated wax is thereafter centrifuged to effect stratification of the wax and separate discharge of the wax and diluted oil phases from the centrifugal rotor.

In the practice of a process of this character, attempts have heretofore been made to employ centrifugal rotors of the familiar type having a so-called disc-bloc; i. e., a series of nested, closely spaced frusto-conical discs affording a plurality of superimposed stratifying chambers from which the wax passes outwardly beyond the outer circumference of the disc-bloc and the oil passes inwardly beyond the inner circumference, the wax and oil being discharged over separate weirs or ring-dams which control the desired balance of the material discharged from the wax outlet against the constituents in the main body of the centrifugal rotor. In the separation of substances from each other in separators of this type, the mixture which is subjected to centrifugation is commonly fed to an intermediate zone of the rotor between the outer and inner circumferences of the discs and is passed longitudinally of the rotor through openings in the discs at that zone to points between the discs within which stratification occurs. In attempted separation of wax from diluted petroleum stock by such a process, difficulty is frequently encountered because of the fact that the wax clogs the spaces between successive stratifying discs and thus renders continuous centrifugation over a substantial period of time difficult or impossible. An object of the present invention has been to solve this problem.

In the process described in the patent to P. T. Sharples No. 1,351,265, a carrier liquid which is immiscible with the oil and wax is employed to facilitate flow of the wax phase through the centrifugal rotor and discharge of wax from the rotor. This carrier liquid fulfills the important function of preventing stickage of wax to parts of the rotor. It also facilitates flow of the wax circumferentially of the rotor to the points from which it is discharged from the rotor. An improvement invented by the present applicant was later used in the practice of the process of Patent No. 1,351,265, and this improvement is described and claimed in the patent to Jones No. 1,649,117. It consists in the feed of the carrier liquid to the centrifugal rotor adjacent the point of discharge of wax therefrom. An important advantage of this improvement consists in the fact that it enables the operator to employ carrier liquid in a heated condition, thereby melting the wax and preventing stickage thereof in the apparatus into which it is discharged from the rotor. If an attempt were made to feel hot carrier liquid together with the oil into the centrifugal rotor, this carrier liquid would re-melt much of the wax precipitated by the preceding chilling operation and thereby cause re-solution of this wax in the oil phase, resulting in discharge of this dissolved wax with the oil and consequent inadequate dewaxing. By feeding the carrier liquid at the point of wax

discharge, it is possible to employ hot carrier liquid, since the hot carrier liquid does not in this case come into contact with the wax until the wax has been separated from the oil layer by stratification.

Wax discharged from the centrifugal separator in any centrifugal dewaxing operation contains a substantial quantity of entrained oil, the oil content of the wax varying between 20 and 50%. It is, of course, desirable to reclaim this entrained oil in case this can be done economically. A particular object of the present invention has been to afford a process whereby the oil entrained in the wax can be economically recovered.

A still further object of the invention consists in the development of a process in which the wax stratified from the oil in the main body of the rotor is supported on an auxiliary spacing liquid which lies adjacent to the inner circumference of the rotor wall and prevents adhesion of wax to that wall, but in which that spacing liquid is not discharged with the wax and does not therefore contaminate the discharged wax.

In cases in which hot carrier liquid is discharged with the wax, the carrier liquid must be separated from the wax by somewhat elaborate procedure before an attempt is made to treat the wax to separate further oil therefrom. This procedure involves separating the bulk of the carrier liquid by gravity settling and partial distillation to evaporate the last traces of carrier liquid. In case a procedure of this type is adopted, the wax must also be diluted and re-chilled to re-precipitate the wax prior to the second step of centrifugation. A particular feature of the present invention consists in the performance of the dewaxing operation in such a way as to avoid pollution of the separated wax with carrier liquid, and to discharge the wax in dilution in a chilled liquid which can be advantageously used in the second step of centrifugation employed to remove entrained oil from the wax. A further feature of the invention consists in the discharge of wax in such dilution in a chilled state, thereby avoiding expensive additional chilling to re-precipitate the wax prior to the second centrifuging step.

Further objects and advantages of the invention will be evident from a reading of the following specification in the light of the attached drawing, in which the single figure is a cross-sectional view of a centrifugal separator embodying the mechanical features of the invention and in which the process features of the invention may be practiced.

Referring to the drawing by reference characters, the rotor 10 of the centrifugal separator is driven by suitable means such as the shaft 28 illustrated in the drawing. This rotor comprises a main body portion 11 and a top portion 12 secured to the main body portion in conventional manner by a clamping ring 13. In the preferred embodiment of the invention, a disc-bloc 14, consisting of a plurality of nested frusto-conical stratifying discs, is secured in position within the rotor between the top portion 12 and the main body portion 11.

A tube 15 may be secured to the interior of the rotor and receives from the feed conduit 16 the material to be treated. The tube 15 communicates with the main body of the rotor through a plurality of openings 29 adjacent the base of the rotor, these openings directing the mixture under treatment into the space 26 surrounding the outer circumference 25 of the disc-bloc. The rotor is provided with a plurality of discharge outlets 17 communicating with the outer portion of the separating space 26 and adapted to receive the heavy effluent from the rotor after that effluent has passed around the outer circumference of the frusto-conical disc 18 which constitutes the dividing wall for separating and separately discharging the light and heavy effluents respectively. Material passing upwardly through outlet 17 passes around ring-dam 19 into a cover 20 of conventional design. The light effluent from the rotor passes inwardly between the respective discs of the disc-bloc 14 and is discharged through space 21 communicating with the inner circumference of the disc-bloc, into the light effluent receiving cover 22.

A conduit 23 is provided between the covers 20 and 22 and conveys liquid to a nozzle 24 similar to that described in the patent to Jones No. 1,649,117, and this liquid is thereafter directed into the centrifugal rotor adjacent the point of discharge of wax effluent from the rotor over ring-dam 19, just as in the case of the applicant's prior Patent 1,649,117.

The bottom of the upper cover 22 may be separated from the top of the lower cover 20 by an insulating air space, thereby preventing undesired heat transfer between cover 20 and cover 22.

The advantages of the above described centrifugal separator and the nature of the process of the present invention will be obvious from the following description of that process in the illustrative embodiments discussed hereinafter.

In the practice of the process of the invention using the machine described above, a mixture containing oil diluted with naphtha, hexane, or other diluent of sufficiently low specific gravity and in sufficient quantity to render the oil phase substantially lighter than the precipitated wax phase, and containing precipitated wax, is fed through conduit 16 into the feed tube 15 of the rotor. This mixture passes outwardly through openings 29 into the stratifying space 26. In accordance with the preferred embodiment of the invention, this space 26 is relatively large, the discs of disc-bloc 14 being relatively small in diameter, and terminating a considerable distance radially within the zone occupied by the plastic wax layer which separates from the oil solution. It will thus be seen that, in this embodiment, the principal stratifying action of the rotor occurs in the space 26, which lies entirely outwardly of the disc-bloc. Oil passing inwardly into the disc-bloc has therefore been deprived of the major part of its wax, and the wax content of such oil consists entirely of small quantities of finely divided wax which have not been removed incident to the stratifying action which takes place in the outer circumferential zone 26. By reason of this fact, the cloggage of discs by wax, such as heretofore occurred in the attempted use of a disc-bloc in centrifugal dewaxing is avoided. The disc-bloc is used only to separate the last traces of wax from oil and deliver into the space 21 and cover 22 a throughly dewaxed oil. Since only small quantities of wax pass outwardly from the disc-bloc into the space 26, cloggage is avoided.

The wax which separates as an outer stratum in the outer part of the space 26 passes upwardly around the outer edge of the dividing wall 18 and is discharged through passages 17 and around ring-dam 19 into the cover 20, this wax being

balanced against the concentric strata of wax and oil in the main body of the rotor.

In the preferred embodiment of the invention, a softening agent for the wax is fed through the conduit **23** and nozzle **24**. The softening agent used for this purpose should be miscible with the oil and of lower specific gravity than the wax itself. This softening agent may be preliminarily chilled to approximately the temperature of the wax phase, and may be a light hydrocarbon such as hexane or naphtha, or it may be any liquid capable of softening the wax and preventing adhesion thereof to the parts of the cover **20**. By adding this softening agent in a sufficient proportion to effect the desired softening and avoid stickage at these points it has been found possible to perform the dewaxing operation without the use of an aqueous carrier liquid and consequent discharge of the carrier liquid with the wax. A part of the diluent may also be separately introduced into the cover **20** to prevent stickage of wax to this cover.

A further feature of the invention consists in the provision of an auxiliary spacing liquid which prevents adhesion of wax to the inner circumferential wall of the rotor and facilitates flow of wax circumferentially around the rotor to the discharge points, but is not discharged with the wax. Thus, a liquid such as water or brine may be introduced into the centrifugal rotor in sufficient quantity to form a thin spacing layer of liquid without the wall of the centrifugal rotor. This layer prevents cohesion of wax to the rotor wall, but it is sufficiently thin so that it does not extend inwardly as far as the outer circumference of the dividing wall **18**, and does not therefore prevent continuous discharge of wax from the rotor. This feature of the use of a layer of spacing liquid may be advantageously used in conjunction with the feed of a non-aqueous softening liquid to the upper end of the rotor to assist in discharge of wax over the ring-dam **19**, as described above.

The use of a spacer liquid in this manner has the important advantage that it avoids contamination of discharged wax with auxiliary liquid used to prevent stickage of wax to the rotor wall. Proposals have been made heretofore, as for example, in my prior Patent 1,649,117, to discharge carrier liquid separately from wax which is floated on such carrier liquid in the centrifugal rotor. In accordance with these prior proposals, however, the carrier liquid was continuously discharged from the rotor under triple liquid balance against the oil and wax phases Such triple liquid balance requires very delicate adjustment of the weirs of the centrifugal rotor, however, and the utilization of this triple balance discharge feature has not been adopted commercially. When the triple liquid balance discharge feature is employed, the carrier liquid will, of course, discharge from the centrifugal rotor at the same rate at which it is fed. In the practice of the present invention, on the other hand, the static spacing liquid is retained within the rotor.

In the practice of the present invention, the spacer liquid initially introduced into the centrifugal rotor is maintained as a static layer surrounding the wax phase in the main body of the centrifugal rotor. In connection with the maintenance of this layer of spacer liquid in the centrifugal rotor, certain special problems are encountered, and one of the particular objects of the invention has been to solve these problems. Thus, in the dewaxing of petroleum stocks by separate discharge of the oil and wax phases into the covers **20** and **22**, respectively, employing a spacer liquid surrounding the wax phase, no difficulty is encountered in the initial stages of the operation in connection with the discharge of both oil and wax from the rotor. But the mixture of oil and wax fed to the rotor sometimes contains small particles of ice. These particles of ice are melted because of the heat generated by friction in the rotor, and, therefore, move toward the circumference of the rotor, being of higher specific gravity than the wax phase. The static layer of water or brine solution forming the spacer liquid therefore gradually becomes deeper during the continued use of the rotor in dewaxing. If this water were to be allowed to continue to accumulate in the rotor, it would ultimately attain a depth at which it would contact the outer circumference of the dividing wall **18** and thus seal off the wax discharge outlets **17** of the rotor. Thus, if no special means were provided to avoid accumulation of water in the rotor, the period of time during which the rotor could be continuously employed would be limited by the accumulation of water within the rotor.

In the practice of the invention, the defect above noted is avoided by providing a special outlet for water which accumulates during the course of the dewaxing operation. Thus, a plurality of discharge tubes **30** of restricted capacity communicate at their inner ends with the space beyond the outer circumference of the dividing wall **18**. Each of these tubes terminates in a replaceable discharge nozzle **31**, which is preferably considerably more restricted than even the tubes **30**. The capacity of the tubes **30** and nozzles **31** is more than sufficient to permit water, separated from the material fed to the rotor, to be discharged through these tubes and nozzles as fast as it separates. They are, however, sufficiently restricted to prevent any substantial proportion of the wax from passing through these discharge outlets.

During the continuous use of the machine the stratum of spacer liquid will vary in depth from the inner circumference of the rotor wall to a point just outside of the outermost ends of the tubes **30** and a point at which the inner circumference of the water stratum just reaches the tubes **30**. At times when the stratum of water does not quite reach the outer ends of the tubes **30**, a small amount of wax will be discharged through those tubes. When the stratum of water reaches the tubes **30**, however, a very small amount of water will be discharged through those tubes, as precipitated in the centrifugal rotor, and into the cover **32**. The initial body of carrier liquid is thus maintained as a static body surrounding the wax stratum, and is prevented from accumulating to a point within the outer ends of the tubes **30**.

It is to be noted that an operation of this character is very different from an operation in which triple liquid balance discharge is employed, for no adjustment of the innermost point over which the water discharges during its passage through the tubes **30** is required to take account of differences in specific gravity of oil and wax phases normally encountered in the process of dewaxing.

The injection of a hydrocarbon softening agent

adjacent the zone of discharge of wax over ring-dam 19 has an additional advantage in connection with further processing of the wax by centrifugation or otherwise to remove entrained oil. In prior art practice in which a hot carrier liquid was employed, the use of such liquid introduced two factors of expense into any process designed to recover entrained oil from the wax. In the first place, the carrier liquid, being hot, heated the wax, and the wax therefore required further chilling to precipitate it before being re-centrifuged. In the second place, the carrier liquid had to be removed before such re-chilling and re-running, thereby causing expense incident to this removal step.

When a softening liquid such as naptha or hexane, which is miscible with the oil entrained in the wax, is introduced through the nozzle 24, all of these items of expense are avoided. In the first place, the hydrocarbon or other liquid added through the nozzle 24 is preferably at approximately the same temperature as the wax, and it does not therefore melt the wax. In the second place, this liquid acts as a very desirable diluent in the performance of the ensuing operation of centrifugation to remove further oil and diluent from the wax. The addition of a diluent prior to the re-centrifuging of the wax is desirable in any case, and the amount of softening agent which is added through the nozzle 24 is usually considerably less than the amount of such diluent actually required in order to re-centrifuge the mixture of oil and wax discharging over the ring-dam 19. In view of the fact that the mixture discharged from the cover 20 is already at approximately the same temperature as that of the mixture introduced into the feed tube 15, the wax is in a precipitated condition in the hydrocarbon softening agent oil solution discharged from cover 20, and this mixture therefore requires no further chilling, or at least only slight further chilling prior to the second step of centrifugation by which further oil is recovered from the wax.

Modifications will be obvious to those skilled in the art, and I do not therefore wish to be limited except by the scope of the sub-joined claims.

I claim:

1. The process of dewaxing a mixture which consists of an oil solution containing precipitated wax of higher specific gravity than said oil solution, which comprises introducing the mixture initially into one end of an external substantially unobstructed annular first zone of a centrifugal rotor having a volume at least sufficient to accommodate the entire separated wax; sedimenting wax from said mixture while in said first zone; passing the oil solution from which most of the wax has been separated to an internal second zone immediately contiguous said first zone wherein stratification discs assist separation of residual wax; withdrawing the oil solution from a locality near the center of the second zone; continuously passing the separated wax longitudinally through said unobstructed annular first zone surrounding said internal second zone and withdrawing the separated wax from near the outer periphery of the end of said first zone opposite to the end of introduction of mixture thereto, and introducing a softening agent into contact with said separated wax to assist in discharge of said wax from the centrifugal apparatus.

2. The process of dewaxing a mixture which consists of an oil solution containing precipitated wax of higher specific gravity than said oil solution, which comprises passing said solution to a centrifugal rotor, centrifugally stratifying wax from said oil solution in a zone of said rotor free of stratifying discs to form a plastic wax stratum lying entirely within said unobstructed zone, maintaining a stratum of spacing liquid between said stratum of wax and the inner circumferential wall of the rotor, passing the oil solution from which wax has been removed by said first-mentioned stratifying action inwardly to a disc-bloc zone of said rotor, subjecting said oil solution to further centrifugation within said disc-bloc zone to remove residual wax therefrom, continuously discharging oil solution from the rotor, and continuously and separately discharging wax from said wax stratum away from said spacing liquid and from said rotor.

3. The process of dewaxing a mixture which consists of an oil solution containing precipitated wax of higher specific gravity than said oil solution, which comprises passing said solution to a centrifugal rotor, centrifugally stratifying wax from said oil solution in a zone of said rotor free of stratifying discs to form a plastic wax stratum lying entirely within said unobstructed zone, maintaining a stratum of spacing liquid between said stratum of wax and the inner circumferential wall of the rotor, passing the oil solution from which wax has been removed by said first-mentioned stratifying action inwardly to a disc-bloc zone of said rotor, subjecting said oil solution to further centrifugation within said disc-bloc zone to remove residual wax therefrom, continuously discharging oil solution from the rotor, continuously and separately discharging wax from said wax stratum away from said spacing liquid and from said rotor, and introducing a liquid softening agent of lower specific gravity than said wax into said rotor at a zone adjacent the zone of discharge of wax therefrom.

4. The process of dewaxing a mixture which consists of an oil solution containing precipitated wax of higher specific gravity than said oil solution, which comprises stratifying the wax from said oil solution in a centrifugal rotor, maintaining a static stratum of spacing liquid between said stratum of wax and the inner circumferential wall of the rotor, discharging said oil solution from said centrifugal rotor, separately discharging wax from the wax stratum away from said spacing liquid and from said rotor, and separately discharging as a third effluent liquid stratifying from said wax stratum into said spacing liquid stratum, the discharge of liquid separated from said wax stratum into said spacing liquid stratum being accomplished by passing said liquid through outlets which are restricted to such an extent that only a small proportion of the material of the wax stratum can be discharged through said outlets, but sufficiently large to permit discharge of all of said liquid which stratifies into said spacing liquid stratum therethrough.

5. The process of separating into its constituents a mixture which consists of a suspension of solid material in a liquid of lower specific gravity than said solid constituent which comprises stratifying the solid constituent from said liquid in a centrifugal rotor, maintaining a static stratum of spacing liquid between said stratum of solid constituent and the inner wall of the rotor, discharging said liquid constituent from said centrifugal rotor, separately discharging

said solid constituent away from said spacing liquid and from said rotor, and separately discharging as a third effluent material stratified from said solid constituent stratum into said spacing liquid stratum, the discharge of material separated from said solid constituent stratum into said spacing liquid stratum being accomplished by passing said material through outlets which are restricted to such an extent that only a small proportion of the stratum of solid material can be discharged through said outlets, but sufficiently large to permit the discharge through said outlets of all of the material which stratifies from said solid material stratum into said spacing liquid stratum.

6. The process of separating into its constituents a mixture which consists of a suspension of solid material in a liquid of lower specific gravity than said solid constituent which comprises passing said mixture to a centrifugal rotor, centrifugally stratifying said solids from said liquids in a zone of said rotor free of stratifying discs to form a stratum of solids lying entirely within said unobstructed zone, maintaining a stratum of spacing liquid between said stratum of solids and the inner circumferential wall of the rotor, passing the liquid from which solids have been removed by said first-mentioned stratifying action inwardly to a disc-bloc zone of said rotor, subjecting said liquid to further centrifugation within said disc-bloc zone to remove residual solids therefrom, continuously discharging liquid from the rotor, and continuously and separately discharging solids from said stratum of solids away from said spacing liquid and from said rotor.

7. A process as defined in claim 6, in which a liquid softening agent is introduced into contact with the separated solids in order to assist in discharge of said solids from the centrifugal apparatus.

8. The process of dewaxing a mixture which consists of an oil solution containing precipitated wax of higher specific gravity than said oil solution, which comprises passing said solution to a centrifugal rotor, centrifugally stratifying wax from said oil solution in a zone of said rotor free of stratifying discs to form a plastic wax stratum lying entirely within said unobstructed zone, maintaining a stratum of spacing liquid between said stratum of wax and the inner circumferential wall of the rotor, passing the oil solution from which wax has been removed by said first-mentioned stratifying action inwardly to a disc-bloc zone of said rotor, subjecting said oil solution to further centrifugation within said disc-bloc zone to remove residual wax therefrom, continuously discharging oil solution from the rotor, continuously and separately discharging wax from said wax stratum away from said spacing liquid and from said rotor, and introducing a liquid softening agent into contact with said separated wax to assist in discharge of said wax from the centrifugal apparatus.

9. The process of dewaxing a mixture which consists of an oil solution containing precipitated wax of higher specific gravity than said oil solution, which comprises introducing the mixture initially near one end of an external substantially unobstructed annular first zone of a centrifugal rotor having a volume at least sufficient to accommodate the entire separated wax; sedimenting wax from said mixture while in said first zone; passing the oil solution from which most of the wax has been separated to an internal second zone immediately contiguous said first zone wherein stratification discs assist separation of residual wax; withdrawing the oil solution from a locality near the center of the second zone; continuously passing the separated wax longitudinally through said unobstructed annular first zone surrounding said internal second zone and withdrawing the separated wax from near the outer periphery of the other end of said first zone, and introducing a softening agent into contact with said separated wax to assist in discharge of said wax from the centrifugal apparatus.

LEO D. JONES.

CERTIFICATE OF CORRECTION.

Patent No. 2,266,554. December 16, 1941.

LEO D. JONES.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 1, first column, lines 32 and 33, for the name "D. T. Sharples" read --P. T. Sharples--; second column, line 48, for "feel" read --feed--; page 2, second column, line 66, for "throughly" read --thoroughly--; page 3, first column, line 33, for "without" read --within--; page 4, first column, line 16, for "naptha" read --naphtha--; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 10th day of March, A. D. 1942.

(Seal)

Henry Van Arsdale,
Acting Commissioner of Patents.