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PROCESS OF DEWAXING HYDROCARBON OILS

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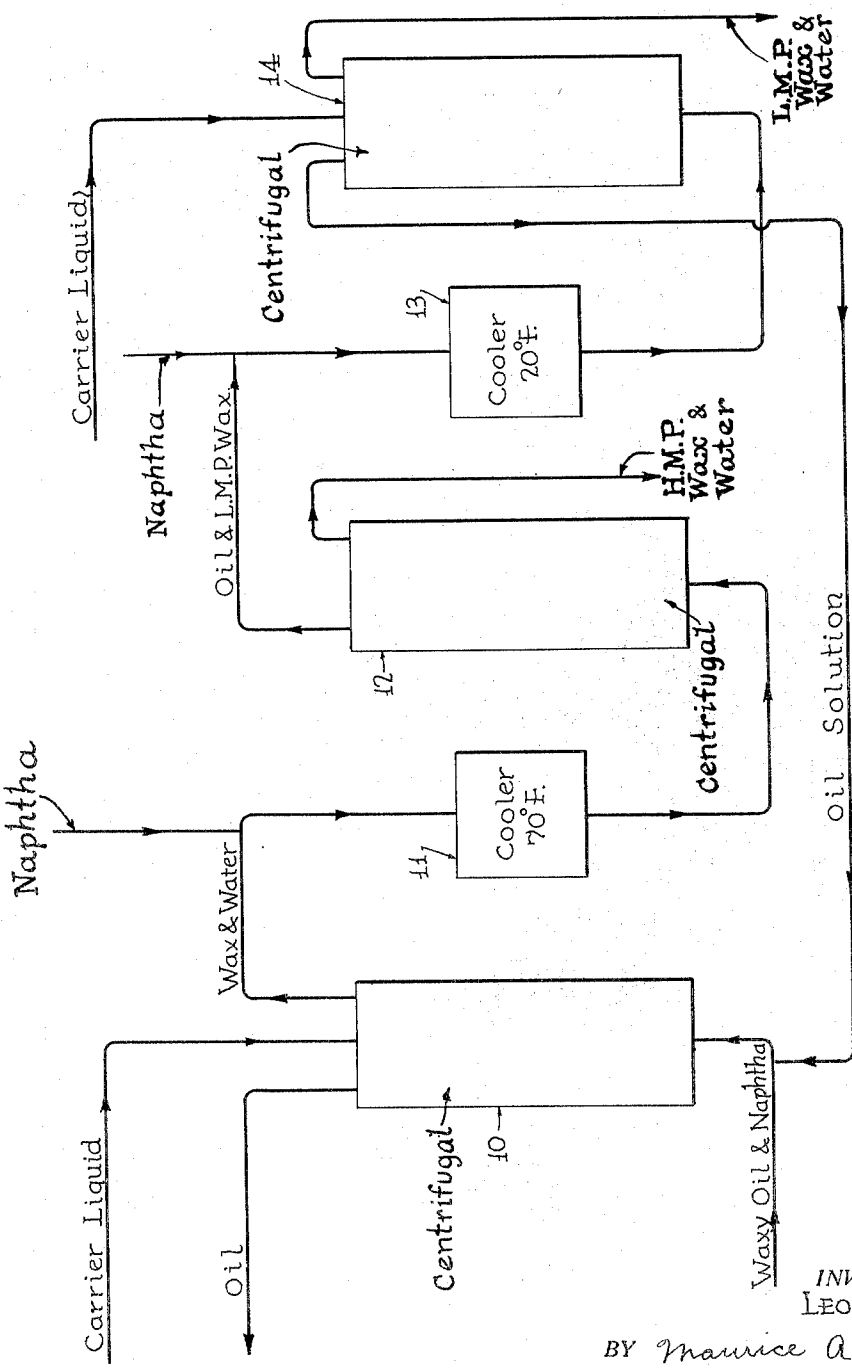

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PROCESS OF DEWAXING HYDROCARBON OILS

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12 Claims. (Cl. 196—18)

The present invention relates to the separation from each other of wax and lubricating oil contained in petroleum stocks. It pertains more particularly to an amplification of the procedure of my prior Patent 1,649,117, whereby the yield of dewaxed oil produced by the process of that patent may be materially increased and a wax of high melting point separately and economically obtained as a product of the process.

In the process of dewaxing described in the patent to P. T. Sharples, 1,351,265, the stock to be dewaxed is first diluted with a sufficient amount of a relatively low-boiling hydrocarbon solvent, such as naphtha, 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 oil and diluent. The mixture of precipitated wax in the oil solution is thereafter passed continuously into a centrifugal to effect stratification of the wax and separate discharge of the wax and diluted oil phases from the centrifugal rotor. In the process described in Sharples Patent 1,351,265, a carrier liquid which is immiscible with the oil and wax is employed to facilitate the flow of the wax phase through the centrifugal rotor and discharge of wax from the rotor. This carrier liquid was originally introduced into the centrifugal rotor at a zone adjacent to the zone of introduction of oil and wax and fulfilled the important function of preventing stickage of wax to parts of the rotor, and reduction of friction in the wax discharge passages.

The process of Sharples Patent 1,351,265 went into extensive commercial use. A disadvantage of the process lay in the fact that the carrier liquid had to be chilled to as low a temperature as that of the chilled oil solution and that difficulty was sometimes encountered in removing wax from the covers into which said wax was discharged from the centrifugal rotor and in effecting flow of this wax through the conduits into which it passed from such discharge covers.

These difficulties in the process of Sharples Patent 1,351,265 were solved by an improvement invented by the present applicant. This improvement is described and claimed in the patent to Jones, 1,649,117. It consisted in feeding the carrier liquid to the upper end of the centrifugal rotor adjacent the point of discharge of wax from the rotor. By feeding carrier liquid adjacent the wax discharge, instead of to the zone of the rotor at which the oil and wax were introduced, the temperature of the carrier liquid had no effect on the wax separation in the rotor. It was then possible, by the application of this top feed principle, to use a heated carrier liquid and melt the discharging wax, thus facilitating the subsequent handling of the wax-carrier liquid mixture.

The present invention relates to a process of effecting further treatment of the wax phase discharged from the centrifugal rotor in accordance with the process of Patent 1,649,117. Wax discharged from the centrifugal separator in the operation of the process of Patent 1,649,117 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 if 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.

When hot carrier liquid is discharged with the wax, in accordance with the process of Patent 1,649,117, the carrier liquid must be separated from the wax by somewhat elaborate procedure in accordance with the prior art 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 subsequent 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 rechilled to reprecipitate the wax prior to the second step of centrifugation to recover the entrained oil.

A particular object of the present invention consists in the provision of an economical procedure whereby the steps of gravity settling and partial distillation to remove carrier liquid from the wax discharged from the primary centrifuge may be avoided, and whereby a wax of high melting point may be produced as a separate by-product in the practice of the invention.

Further objects and advantages of the invention and the manner in which they have been attained will be obvious after a reading of the following specification in the light of the attached flow sheet, in which the single figure represents an illustrative embodiment of the practice of the invention.

Referring to the drawing by reference characters, a mixture of oil and wax, which has been diluted with naphtha and chilled to a temperature below 0° F. to precipitate the wax after dilution, in accordance with the procedure described in Patents 1,351,265 and 1,649,117, referred to

above, is passed to a centrifugal rotor 10. A hot, aqueous carrier liquid is separately passed to the centrifugal rotor 10 adjacent the zone of discharge of wax therefrom. A layer of aqueous carrier liquid occupies the zone adjacent the inner peripheral wall of the rotor 10 and prevents stickage of wax to the wall of the rotor and facilitates the discharge of wax from the rotor, just as in the case of my prior Patent 1,649,117. Dewaxed oil solution is discharged as the lighter effluent from the centrifugal rotor, and a mixture of wax and the carrier liquid is separately discharged as the heavier effluent.

The mixture of wax and carrier liquid (which may be water or an aqueous salt solution) discharged from the rotor 10 is next mixed with a large quantity of a light hydrocarbon diluent, which may be the same diluent (naphtha) used in the primary centrifuging step performed in the rotor 10. The amount of naphtha added at this stage of the process may be from 2⅓ to 19 times as great as the quantity of wax phase discharged from the rotor 10. The mixture produced may, for example, contain 90% of naphtha and 10% of the mixture of wax and oil discharged from the rotor 10. This mixture is next cooled to a temperature substantially higher than the freezing point of water.

If an attempt were made to cool the mixture to a point below the freezing point of the water which it contains, the formation of crystals of ice in the mixture would act as an impediment to subsequent centrifugal separation. If subsequent centrifugal separation were accomplished at a point below the freezing point of the water, the wax separated from this secondary centrifugal separating step would also be of relatively low melting point.

A particular feature of the invention consists in the cooling of the mixture only to a point substantially above the freezing point of water at this stage of the process. By operating in this way, we obtain a wax effluent from the secondary centrifuging operation which is very valuable because of its high melting point, and we also separate water from the oil contained in the wax effluent of the centrifuge 10 in an economical manner.

In a preferred form of the invention, practiced when a separate wax fraction having a melting point of approximately 160° F. is desired, the mixture of naphtha, wax and water formed by diluting with naphtha the wax and water effluent from the centrifuge 10 is passed to a cooler 11, wherein its temperature is reduced to approximately 70° F. This entire mixture is then passed to a secondary centrifugal rotor 12. The water of the mixture, being the heaviest constituent, stratifies adjacent the inner centrifugal wall of the rotor, and the wax and a part of this water are discharged from the rotor together, the water acting as a carrier liquid in accordance with the process of Patent 1,351,265, to prevent adhesion of the wax to the wall of the centrifugal rotor. At the relatively high temperature used in the secondary centrifuging operation, the low melting constituents of the wax are retained in solution in the oil and naphtha.

This solution is separately discharged from the centrifuge 12 and is subjected to a further step of addition of naphtha, chilling and centrifugal separation to remove the wax from the oil and afford a further yield of oil of desirably low pour point. As illustrated in the flow sheet, the mixture of oil and wax, containing the major part of the naphtha introduced prior to the passage of the mixture to the cooler 11, is passed to a second cooler 13, which chills the solution to a temperature sufficiently low to precipitate practically all of the wax from solution; e. g., to a temperature of −20° F.

The mixture of precipitated wax in the solution of oil in naphtha is passed from the cooler 13 to a third centrifugal separator 14. A carrier liquid is separately passed to this third centrifugal separator, preferably in a heated condition, in accordance with Patent 1,649,117. The wax is discharged, together with the carrier liquid, from this third centrifugal, and the solution of oil in naphtha is separately discharged from this centrifugal as the lightest effluent. This oil solution discharged from centrifugal 14 may be subjected to distillation to remove the diluent naphtha therefrom, but this solution can be most economically processed in accordance with the invention by employing it to dilute a fresh charge of oil to be dewaxed in the centrifuge 10. The oil solution discharged from the centrifuge 14 is in a chilled condition, and is therefore particularly suitable for use in diluting the fresh charge of oil to be dewaxed. It also contains a very large proportion of naphtha (e. g., 95%, in case the naphtha added to the mixture of wax and water discharged from centrifuge 10 is added in a ratio to the wax effluent of approximately 9:1), and the presence of this high proportion of naphtha makes the oil effluent from centrifuge 14 a particularly desirable composition for dilution of a fresh charge of oil and wax.

While specific temperatures and specific proportions of diluents have been suggested above, it will be understood that these suggestions are made by way of illustration and not by way of limitation. Thus, the amount of naphtha added to the mixture of wax and water discharged from the centrifuge 10 may vary within fairly wide limits. Similarly, the temperature to which the mixture is chilled in the cooler 11 may be any temperature above the freezing point of water (or other aqueous carried liquid such as brine). In case it is desired to produce a wax effluent from the centrifuge 12 having a melting point of 150° F., for example, the mixture in the cooler 11 may be chilled to a temperature of 40° F. If, on the other hand, it is desired to produce a wax of extremely high melting point (e. g. 170° F.), the mixture may be cooled only to a temperature of 100° F. The temperature to which the oil is chilled at this stage will, of course, determine the relative proportions and character of the wax which remains in solution and the wax which is precipitated respectively, and will accordingly determine the melting point of the wax discharged from the centrifuge 12.

Further 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 subjoined claims.

I claim:

1. In the separation of wax and oil from each other, the process comprising, feeding a mixture of oil and precipitated wax to a centrifugal rotor, feeding a hot aqueous carrier liquid of higher specific gravity than either the oil or wax to said centrifugal rotor, subjecting the mixture to centrifugal stratification within said rotor, discharging oil as the lighter phase from said rotor, discharging wax containing some entrained oil together with said carrier liquid as the heavier phase from the rotor, adding a diluent of lower specific gravity than said wax to the mixture of

wax and carrier liquid discharged from said rotor, passing the mixture so produced to a second centrifugal rotor at a temperature above the freezing point of the carrier liquid and sufficiently high to maintain a substantial part of the wax in solution, but sufficiently low to maintain a higher melting part of the wax content of the mixture in precipitated form, subjecting the mixture to centrifugal stratification in said second centrifugal rotor, discharging oil, containing a part of the wax in solution, as the lighter phase from said second centrifugal rotor, and discharging wax of high melting point together with said carrier liquid as the heavier phase from said second centrifugal rotor.

2. In the separation of wax and oil from each other, the process comprising, feeding a mixture of oil and precipitated wax to a centrifugal rotor, feeding a hot aqueous carrier liquid of higher specific gravity than either the oil or wax to said centrifugal rotor, subjecting the mixture to centrifugal stratification within said rotor, discharging oil as the lighter phase from said rotor, discharging wax containing some entrained oil together with said carrier liquid as the heavier phase from the rotor, adding a diluent of lower specific gravity than said wax to the mixture of wax and carrier liquid discharged from said rotor, chilling the mixture so produced to a temperature above the freezing point of the carrier liquid and sufficiently high to maintain a substantial part of the wax in solution, but sufficiently low to precipitate a higher melting part of the wax content of the mixture, passing the chilled mixture to a second centrifugal rotor, subjecting the mixture to centrifugal stratification in said second centrifugal rotor, discharging oil, containing a part of the wax in solution, as the lighter phase from said second centrifugal rotor, and discharging wax of high melting point together with said carrier liquid as the heavier phase from said second centrifugal rotor.

3. In the separation of wax and oil from each other, the process comprising, feeding a mixture of oil and precipitated wax to a centrifugal rotor, feeding a hot aqueous carrier liquid of higher specific gravity than either the oil or wax to said centrifugal rotor, subjecting the mixture to centrifugal stratification within said rotor, discharging oil as the lighter phase from said rotor, discharging wax containing some entrained oil together with said carrier liquid as the heavier phase from the rotor, adding a diluent of lower specific gravity than said wax to the mixture of wax and carrier liquid discharged from said rotor, chilling the mixture so produced to a temperature between 40 and 100° F., passing the chilled mixture to a second centrifugal rotor, subjecting the mixture to centrifugal stratification in said second centrifugal rotor, discharging oil, containing a part of the wax in solution, as the lighter phase from said second centrifugal rotor, and discharging wax of high melting point together with said carrier liquid as the heavier phase from said second centrifugal rotor.

4. In the separation of wax and oil from each other, the process comprising, feeding a mixture of oil and precipitated wax to a centrifugal rotor, feeding a hot aqueous carrier liquid of higher specific gravity than either the oil or wax to said centrifugal rotor, subjecting the mixture to centrifugal stratification within said rotor, discharging oil as the lighter phase from said rotor, discharging wax containing some entrained oil together with said carrier liquid as the heavier phase from the rotor, adding a diluent of lower specific gravity than said wax to the mixture of wax and carrier liquid discharged from said rotor, passing the mixture so produced to a second centrifugal rotor at a temperature above the freezing point of the carrier liquid and sufficiently high to maintain a substantial part of the wax in solution, but sufficiently low to maintain a higher melting part of the wax content of the mixture in precipitated form, subjecting the mixture to centrifugal stratification in said second centrifugal rotor, discharging oil, containing a part of the wax in solution, as the lighter phase from said second centrifugal rotor, discharging wax of high melting point together with said carrier liquid as the heavier phase from said second centrifugal rotor, chilling the solution of wax in oil discharged from said second centrifugal rotor to a temperature sufficiently low to precipitate further wax therefrom, passing the mixture of oil and wax produced by said last-mentioned chilling operation to a third centrifugal rotor, subjecting the mixture to centrifugal stratification in said third centrifugal rotor and separately discharging oil and wax from said third centrifugal rotor.

5. In the separation of wax and oil from each other, the process comprising, feeding a mixture of oil and precipitated wax to a centrifugal rotor, feeding a hot aqueous carrier liquid of higher specific gravity than either the oil or wax to said centrifugal rotor, subjecting the mixture to centrifugal stratification within said rotor, discharging oil as the lighter phase from said rotor, discharging wax containing some entrained oil together with said carrier liquid as the heavier phase from the rotor, adding a diluent of lower specific gravity than said wax to the mixture of wax and carrier liquid discharged from said rotor, chilling the mixture so produced to a temperature above the freezing point of the carrier liquid and sufficiently high to maintain a substantial part of the wax in solution, but sufficiently low to precipitate a higher melting part of the wax content of the mixture, passing the chilled mixture to a second centrifugal rotor, subjecting the mixture to centrifugal stratification in said second centrifugal rotor, discharging oil, containing a part of the wax in solution, as the lighter phase from said second centrifugal rotor, discharging wax of high melting point together with said carrier liquid as the heavier phase from said second centrifugal rotor, chilling the solution of wax in oil discharged from said second centrifugal rotor to a temperature sufficiently low to precipitate further wax therefrom, passing the mixture of oil and wax produced by said last-mentioned chilling operation to a third centrifugal rotor, subjecting the mixture to centrifugal stratification in said third centrifugal rotor and separately discharging oil and wax from said third centrifugal rotor.

6. In the separation of wax and oil from each other, the process comprising, feeding a mixture of oil and precipitated wax to a centrifugal rotor, feeding a hot aqueous carrier liquid of higher specific gravity than either the oil or wax to said centrifugal rotor, subjecting the mixture to centrifugal stratification within said rotor, discharging oil as the lighter phase from said rotor, discharging wax containing some entrained oil together with said carrier liquid as the heavier phase from the rotor, adding a diluent of lower specific gravity than said wax to the mixture of

wax and carrier liquid discharged from said rotor, chilling the mixture so produced to a temperature between 40 and 100° F., passing the chilled mixture to a second centrifugal rotor, subjecting the mixture to centrifugal stratification in said second centrifugal rotor, discharging oil, containing a part of the wax in solution, as the lighter phase from said second centrifugal rotor, discharging wax of high melting point together with said carrier liquid as the heavier phase from said second centrifugal rotor, chilling the solution of wax in oil discharged from said second centrifugal rotor to a temperature sufficiently low to precipitate further wax therefrom, passing the mixture of oil and wax produced by said last-mentioned chilling operation to a third centrifugal rotor, subjecting the mixture to centrifugal stratification in said third centrifugal rotor and separately discharging oil and wax from said third centrifugal rotor.

7. In the separation of wax and oil from each other, the process comprising, feeding a mixture of oil and precipitated wax to a centrifugal rotor, feeding a hot aqueous carrier liquid of higher specific gravity than either the oil or wax to said centrifugal rotor, subjecting the mixture to centrifugal stratification within said rotor, discharging oil as the lighter phase from said rotor, discharging wax containing some entrained oil together with said carrier liquid as the heavier phase from the rotor, adding a diluent of lower specific gravity than said wax to the mixture of wax and carrier liquid discharged from said rotor, passing the mixture so produced to a second centrifugal rotor at a temperature above the freezing point of the carrier liquid and sufficiently high to maintain a substantial part of the wax in solution, but sufficiently low to maintain a higher melting part of the wax content of the mixture in precipitated form, subjecting the mixture to centrifugal stratification in said second centrifugal rotor, discharging oil, containing a part of the wax in solution, as the lighter phase from said second centrifugal rotor, discharging wax of high melting point together with said carrier liquid as the heavier phase from said second centrifugal rotor, chilling the solution of wax in oil discharged from said centrifugal rotor to a temperature below 0° F. to precipitate further wax from solution, passing the mixture of oil and wax produced by said last-mentioned chilling operation to a third centrifugal rotor, subjecting the mixture to centrifugal stratification in said third centrifugal rotor and separately discharging oil and wax from said third centrifugal rotor.

8. In the separation of wax and oil from each other, the process comprising, feeding a mixture of oil and precipitated wax to a centrifugal rotor, feeding a hot aqueous carrier liquid of higher specific gravity than either the oil or wax to said centrifugal rotor, subjecting the mixture to centrifugal stratification within said rotor, discharging oil as the lighter phase from said rotor, discharging wax containing some entrained oil together with said carrier liquid as the heavier phase from the rotor, adding a diluent of lower specific gravity than said wax to the mixture of wax and carrier liquid discharged from said rotor, passing the mixture so produced to a second centrifugal rotor at a temperature above the freezing point of the carrier liquid and sufficiently high to maintain a substantial part of the wax in solution, but sufficiently low to maintain a higher melting part of the wax content of the mixture in precipitated form, subjecting the mixture to centrifugal stratification in said second centrifugal rotor, discharging oil, containing a part of the wax in solution, as the lighter phase from said second centrifugal rotor, discharging wax of high melting point together with said carrier liquid as the heavier phase from said second centrifugal rotor, chilling the solution of wax in oil discharged from said second centrifugal rotor to a temperature sufficiently low to precipitate further wax therefrom, passing the mixture of oil and wax produced by said last-mentioned chilling operation to a third centrifugal rotor, subjecting the mixture to centrifugal stratification in said third centrifugal rotor and separately discharging oil as the lighter phase from said third centrifugal rotor, and discharging wax together with carrier liquid separately added to said third centrifugal rotor as the heavier phase from said third centrifugal rotor.

9. In the separation of wax and oil from each other, the process comprising, feeding a mixture of oil and precipitated wax to a centrifugal rotor, feeding a hot aqueous carrier liquid of higher specific gravity than either the oil or wax to said centrifugal rotor, subjecting the mixture to centrifugal stratification within said rotor, discharging oil as the lighter phase from said rotor, discharging wax containing some entrained oil together with said carrier liquid as the heavier phase from the rotor, adding a diluent of lower specific gravity than said wax to the mixture of wax and carrier liquid discharged from said rotor, passing the mixture so produced to a second centrifugal rotor at a temperature above the freezing point of the carrier liquid and sufficiently high to maintain a substantial part of the wax in solution, but sufficiently low to maintain a higher melting part of the wax content of the mixture in precipitated form, subjecting the mixture to centrifugal stratification in said second centrifugal rotor, discharging oil, containing a part of the wax in solution, as the lighter phase from said second centrifugal rotor, discharging wax of high melting point together with said carrier liquid as the heavier phase from said second centrifugal rotor, chilling the solution of wax in oil discharged from said second centrifugal rotor to a temperature sufficiently low to precipitate further wax therefrom, passing the mixture of oil and wax produced by said last-mentioned chilling operation to a third centrifugal rotor, subjecting the mixture to centrifugal stratification in said third centrifugal rotor and separately discharging oil as the lighter phase from said third centrifugal rotor, and discharging wax together with carrier liquid in a heated condition separately added to said third centrifugal rotor as the heavier phase from said third centrifugal rotor.

10. In the separation of wax and oil from each other, the process comprising, feeding a mixture of oil and precipitated wax to a centrifugal rotor, feeding a hot aqueous carrier liquid of higher specific gravity than either the oil or wax to said centrifugal rotor, subjecting the mixture to centrifugal stratification within said rotor, discharging oil as the lighter phase from said rotor, discharging wax containing some entrained oil together with said carrier liquid as the heavier phase from the rotor, adding a diluent of lower specific gravity than said wax to the mixture of wax and carrier liquid discharged from said rotor, passing the mixture so produced to a second centrifugal rotor at a temperature above the freezing point of the carrier liquid and sufficient-

ly high to maintain a substantial part of the wax in solution, but sufficiently low to maintain a higher melting part of the wax content of the mixture in precipitated form, subjecting the mixture to centrifugal stratification in said second centrifugal rotor, discharging oil, containing a part of the wax in solution, as the lighter phase from said second centrifugal rotor, discharging wax of high melting point together with said carrier liquid as the heavier phase from said second centrifugal rotor, chilling the solution of wax in oil discharged from said second centrifugal rotor to a temperature sufficiently low to precipitate further wax therefrom, passing the mixture of oil and wax produced by said last-mentioned chilling operation to a third centrifugal rotor, subjecting the mixture to centrifugal stratification in said third centrifugal rotor, separately discharging oil and wax from said third centrifugal rotor and diluting fresh stock to be dewaxed with the solution of oil in diluent discharged from said third centrifugal rotor.

11. In the separation of wax and oil from each other, the process comprising, feeding a mixture of oil and precipitated wax to a centrifugal rotor, feeding a hot aqueous carrier liquid of higher specific gravity than either the oil or wax to said centrifugal rotor, subjecting the mixture to centrifugal stratification within said rotor, discharging oil as the lighter phase from said rotor, discharging wax containing some entrained oil together with said carrier liquid as the heavier phase from the rotor, adding a diluent of lower specific gravity than said wax to the mixture of wax and carrier liquid discharged from said rotor, passing the mixture so produced to a second centrifugal rotor at a temperature above the freezing point of the carrier liquid and sufficiently high to maintain a substantial part of the wax in solution, but sufficiently low to maintain a higher melting part of the wax content of the mixture in precipitated form, subjecting the mixture to centrifugal stratification in said second centrifugal rotor, discharging oil, containing a part of the wax in solution, as the lighter phase from said second centrifugal rotor, discharging wax of high melting point together with said carrier liquid as the heavier phase from said second centrifugal rotor, chilling the solution of wax in oil discharged from said second centrifugal rotor to a temperature sufficiently low to precipitate further wax therefrom, passing the mixture of oil and wax produced by said last-mentioned chilling operation to a third centrifugal rotor, subjecting the mixture to centrifugal stratification in said third centrifugal rotor, separately discharging oil and wax from said third centrifugal rotor and diluting fresh stock to be dewaxed with the solution of oil in diluent discharged from said third centrifugal rotor while said solution of oil in diluent is still in a chilled condition.

12. In the separation of wax and oil from each other, the process comprising, feeding a mixture of oil and precipitated wax to a centrifugal rotor, feeding a hot aqueous carrier liquid of higher specific gravity than either the oil or wax to said centrifugal rotor, subjecting the mixture to centrifugal stratification within said rotor, discharging oil as the lighter phase from said rotor, discharging wax containing some entrained oil together with said carrier liquid as the heavier phase from the rotor, passing the wax and carrier liquid discharged from said rotor, together with oil discharged with said wax from said rotor and sufficient diluent to render the oil phase lighter than the precipitated wax, to a second centrifugal rotor at a temperature above the freezing point of the carrier liquid and sufficiently high to maintain a substantial part of the wax in solution, but sufficiently low to maintain a higher melting part of the wax content of the mixture in precipitated form, subjecting the mixture to centrifugal stratification in said second centrifugal rotor, discharging oil containing a part of the wax in solution as the lighter phase from said second centrifugal rotor, and discharging wax of high melting point together with said carrier liquid as the heavier phase from said second centrifugal rotor.

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