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CENTRIFUGAL DEWAXING OF OILS

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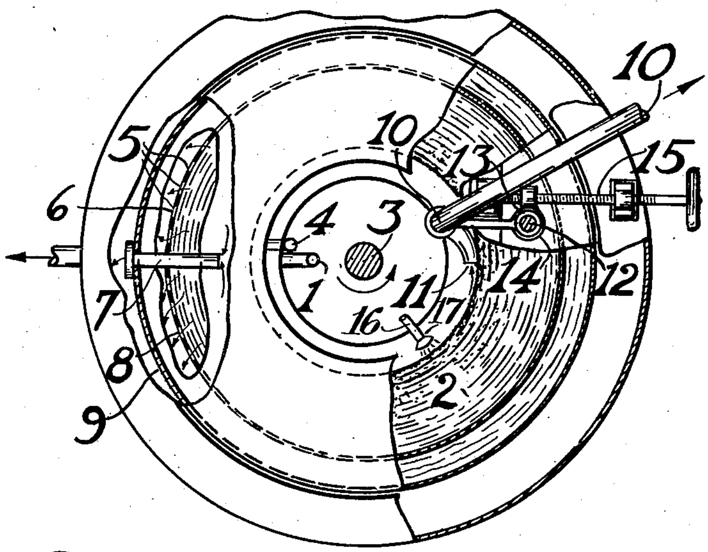

Fig. - 2

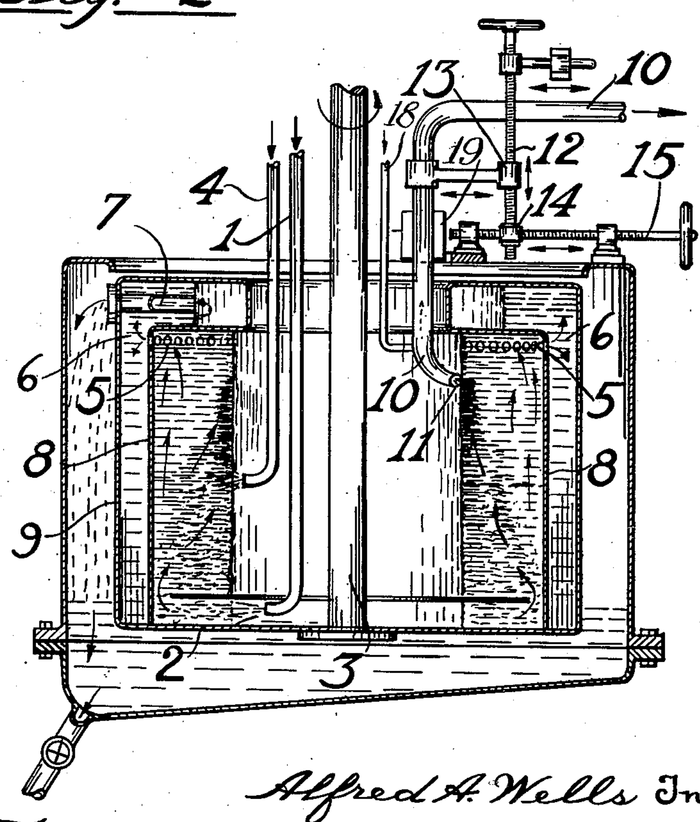

Fig. - 1

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CENTRIFUGAL DEWAXING OF OILS

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13 Claims. (Cl. 196—19)

This invention relates to improved methods for dewaxing oils with a centrifuge and, more particularly, to improved means for removing wax from the centrifuge.

The centrifuge has long been known to be a very effective means for separating wax from waxy petroleum oils. In practice, the waxy oil is usually mixed with a suitable solvent having a preferential solubility for the oil and having little or no solvent action on wax at low temperatures, the mixture is cooled until the wax is precipitated in a solid amorphous or crystalline form and the chilled mixture is then whirled in a centrifuge to concentrate the separated wax in one layer and the oil-solvent solution in a separate layer. A number of satisfactory methods for conducting the operation up to this point are known. The separated oil may be readily removed from the centrifugal zone by causing it to flow out over suitable retaining walls, weirs and the like, or through a semi-permeable membrane, such as a filter blanket, which retains the precipitated wax. Great difficulty, however, has been experienced in removing the wax from the centrifugal zone. The use of flotation agents, such as brine, has made possible the continuous removal of certain types of amorphous waxes under carefully controlled operating conditions. Even this method has been unsatisfactory for the removal of crystalline waxes from the centrifugal zone, as it has not been found possible to cause these waxes to flow over the necessary retaining walls or through passages of the ordinary types used for withdrawal of materials. The use of plows, scoops, screws and the like interferes seriously with the separation desired to be accomplished in the centrifuge. Such methods are, accordingly, applied only to batch operation with resulting loss in capacity and efficiency in operation.

It has now been found that the separated wax can be readily, conveniently and continuously removed from an operating centrifuge by taking advantage of the kinetic energy of the whirling wax. This can be done by opposing the rotation of the wax with an open ended tube or other conduit so that a portion of the wax is continuously separated from the rotating wax layer and is forced by its own momentum to pass into the tube and through the tube out of the centrifuge.

Substantially any type of wax may be removed from a centrifuge by this invention, including the waxes separated from both paraffinic and mixed base crudes, and from residual and distillate products of extraction, hydrogenation, cracking, dehydrogenation and other treatments of such crudes. No difficulty is experienced in removing even the most crystalline types of waxes from a centrifuge with this invention.

The drawing illustrates one type of apparatus suitable for conducting the herein-described invention when using a dewaxing solvent of higher specific gravity than that of the separated wax and will be described in connection with an example illustrating the operation of the apparatus in dewaxing.

The drawing represents a sketch of one type of apparatus suitable for this invention and indicates the flow of materials. Figure 1 is an elevation, partly in section, of a centrifuge embodying this invention. Figure 2 is a plan view, partly in section, of the centrifuge shown in Figure 1.

A waxy lubricating oil containing wax which separates in crystalline form on cooling, is mixed with three volumes of a solvent containing 25% carbon tetrachloride and 75% dichlorethane. The resulting solution is cooled to −17° F., whereupon large amounts of crystalline wax are precipitated. The resulting cold slurry is passed by line 1 into a centrifugal bowl 2 which is, for example, about 24″ in diameter and which is rotated by shaft 3 at about 1,800 R. P. M. The mixture in passing up the bowl separates rapidly into a liquid layer substantially free of precipitated wax and a waxy layer containing substantially all the precipitated wax with more or less occluded liquid. The wax layer may be washed with a suitable solvent, for example the same as that originally mixed with the oil, which is added through tube 4 and sprayed on the surface of the rising wax layer. The separated oil-solvent layer may be withdrawn from the separation zone through suitable openings 5 which are preferably at or near the top of the wall 8 of the centrifugal bowl. This liquid then passes through chamber 6 and out of the centrifuge through tube 7 which is adjusted to maintain a constant level in the centrifugal bowl. It is obvious that many other methods and other modifications of the method illustrated may be used for the withdrawal of this liquid phase, for example, the liquid draw-off tube 7 and the outer wall 9 may be omitted and the desired liquid level maintained by a suitable pressure drop through the openings 5 or a combination of a smaller pressure drop through these openings may be used with tube 7 to obtain smoother operation with elimination of possible surges. All or any part of the wall 8 of the centrifuge may also be made perforate and covered with a suitable filtering medium, such as the filter blankets ordinarily used in filter presses for dewaxing oils.

The precipitated wax travels upwardly and is displaced inwardly because of its lower specific gravity until it forms a wax cake on the inner surface near the top of the rotating mixture. A tube 10 is so placed that its open end 11 faces in a direction opposite to the direction of rotation of the centrifuge and cuts into the wax cake. The edge of the tube cutting into the wax cake is preferably made razor sharp. It is also preferably tapered to a somewhat smaller diameter than the remainder of the tube in order to avoid undue back pressure being exerted on the wax cake by the wax flowing into the tube and to avoid interference with the wax not removed, insofar as possible. Wax is thus severed from the wax cake while traveling at a very high linear velocity, say about 4,000 to 15,000 feet per minute or even faster. The kinetic energy of the severed wax, due to this high velocity, has been found sufficient to cause even stiff crystalline wax, which heretofore has been considered impossible to remove continuously from a centrifuge, to flow readily and continuously through the tube 10 and out of the centrifuge. Pressures of 50 or 75 to 150 pounds per square inch have been observed in the tube 10 near the entrance end, and pressures of 300 pounds per square inch and even higher may be obtained at the higher linear speeds. For example, when operating with 137.6 gallons per hour of the above described mixture, a waxy layer consisting of 34% of the original oil is continuously withdrawn through the tube 10, permitting the continuous withdrawal of the oil-solvent liquid phase, to produce a 66% yield of a dewaxed oil having a pour point of 10° F. from the original oil of 120° F. pour point.

This invention may also be applied to match operation, if desired, thereby making it possible to remove the wax layer during or at the end of each separation period without change in the speed of the centrifuge. The position of the tube 10 may be made adjustable; as by means of a vertical screw 12 operating in a threaded collar 13 attached to the tube, the screw 12 being attached to a second threaded collar 14 through which a horizontal screw 15 operates. Both screws may be maintained in their relative positions by suitable bearings and guides. The horizontal adjustable feature will be found of advantage in removing wax in batch operation of the centrifuge, as the cutting edge of the tube can be moved outwardly as the wax is removed. The vertical adjustable feature is ordinarily not required, but may be of use in the case of very stiff wax cakes which do not flow sufficiently freely in the bowl to make wax removal from a single horizontal plane satisfactory. A particularly effective method of operation in batch operation in which the inner wall 8 of the centrifuge is covered with a filter blanket is to permit the wax cake to build up until the liquid phase has largely passed through the blanket and wax is resting against it, then remove the wax by means of the tube until only a thin layer of wax remains, preferably of less thickness than the radial distance between the overflow level of the tube 7 and the blanket, then add sufficient heavy wash liquid to the bowl to float the wax inwardly from the blanket, and removing the so floating layer of wax with the tube 10.

It is preferable in using this invention to remove the wax layer from the centrifugal bowl while it still contains an appreciable amount of liquid occluded or otherwise admixed with the solid wax particles. The wax layer may contain some of the oil-solvent solution or may have been washed substantially free of lubricating oil with a washing liquid, a portion of the latter being retained. The contained liquid permits the solid particles of wax to slip more easily over one another and hence permits the withdrawal of the wax layer from the centrifuge more readily by means of the herein described invention. While it is possible when operating at sufficiently high linear speeds to remove substantially any wax from a centrifuge by the means described, it is generally desirable that the wax being removed should contain at least 10 to 25% of liquid and it is customary, in practice, for the liquid content of this cake to run even as high as 50 or 75 or 80%. The cake, however, is solid even with these high concentrations of liquid, due to the known ability of wax crystals to interlace and cause the apparent solidification of a wax-oil mixture at a temperature far above the actual freezing point of the oil phase.

The wax cake removed from the centrifuge, as described above, may be mixed with additional solvent and, with or without heating and cooling to effect solution and reprecipitation of the wax, may then be passed to a second centrifuge from which additional oil of low pour point may be recovered and a wax layer may be withdrawn, containing a correspondingly decreased amount of oil, by the same means described above.

In case it is desired to operate the centrifuge so as to obtain a substantially dry wax cake, the fluidity of this wax may be increased by spraying a suitable solvent therefor on the surface of the wax just before it is severed from the cake by the tube described above. This may be done through line 16 provided with spray head 17 which directs a spray of solvent against the wax cake near the point of removal. Such solvent, for example, carbon tetrachloride or naphtha, which may conveniently be at room temperature or other relatively high temperature, may also be added to the wax stream shortly after its entry into the tube as through line 18 or the tube may be heated by any suitable means such as by steam jacket 19 disposed around pipe 10. These provisions will ordinarily be found unnecessary however, if a small amount of liquid is allowed to remain in the wax being separated.

While this process may be conducted with the separated wax layer as either the lighter or the heavier phase, the former method of operation is generally to be preferred, because of the ease of applying the tube for removing the wax to the inner surface of the wax layer. Any suitable dewaxing solvent of higher specific gravity than the separated wax may be used for this operation. Among such solvents may be mentioned illustratively carbon tetrachloride, dichlorethane, dichlorpropane, dichlorethylene, trichlorethylene, tetrachlorethylene, dichlorpropylene and the like. These solvents may be used alone or in combination with each other and/or with other dewaxing solvents of lower specific gravities, such as naphtha, benzol, acetone, the lower aliphatic alcohols and the like. The solvents of lower density are preferably used in sufficiently small amounts so that the total oil-solvent solution is of higher specific gravity than the separated wax.

This invention is not to be limited to any illustrations, examples or explanations which have been presented herein solely for purposes of illustration, but is limited only by the following claims in which it is desired to claim all novelty insofar as the prior art permits.

I claim:

1. Process for dewaxing oil comprising centrifugally separating wax as a solid phase from a liquid oil and withdrawing the separated wax from the whirling zone through a constricted passageway by means of the kinetic energy of the said separated wax.

2. Process for dewaxing oil comprising admixing therewith a solvent of relatively higher specific gravity having preferential solubility for oil in contact with wax, cooling the mixture to cause separation of wax as a solid phase, subjecting the cooled mixture to centrifugal force whereby a lighter waxy layer separates from a heavier oily layer containing a relatively higher content of oil and withdrawing the separated waxy layer from the whirling zone through a constricted passageway by means of its kinetic energy and without the application of force from the materials remaining in the centrifugal zone.

3. Process according to claim 2 in which the said waxy oil is a petroleum lubricating oil.

4. Process according to claim 2 in which the said separated wax is in crystalline form.

5. Process according to claim 2 in which the separated waxy layer contains more than 10% of liquid.

6. Process according to claim 2 in which the separated waxy layer contains more than 25% of liquid.

7. Process according to claim 2 in which the inner level of the separated wax in the whirling zone is maintained substantially constant by maintaining a substantially constant level of the liquid phase therein.

8. Process according to claim 2 in which the fluidity of the separated wax is increased prior to its point of removal.

9. Process according to claim 2 in which the fluidity of the separated wax is increased during its flow in said confined stream.

10. Process according to claim 2 in which the fluidity of the separated wax is increased during its flow in said confined stream by the addition of a suitable solvent for wax thereto.

11. Process according to claim 2 in which the fluidity of said separated wax is increased during its flow in said confined stream by raising the temperature of the wax in said stream.

12. Process for dewaxing a waxy lubricating oil, comprising centrifugally concentrating the waxy component thereof in one layer, in a substantially solid form, and the oily component thereof in a separate liquid layer of different density, continuously severing wax from the separated rotating wax layer, whereby the velocity of the severed wax is not substantially impeded, withdrawing the severed wax from the centrifugal zone by means of the kinetic energy of the said severed wax and without the application of force from the materials remaining in the centrifugal zone.

13. Process for dewaxing oil comprising admixing therewith a solvent of relatively higher specific gravity having preferential solubility for oil in contact with wax, cooling the mixture to cause separation of wax as a solid phase, subjecting the cooled mixture to centrifugal force whereby a lighter waxy layer separates inwardly from a heavier oily layer, continuously severing wax from the separated rotating wax layer whereby the velocity of the severed wax is not substantially impeded, and withdrawing the severed wax inwardly a substantial distance from the inner surface of the rotating wax layer by means of the kinetic energy of the severed wax and without the application of force from the materials remaining in the centrifugal zone.

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