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Description

Background of the invention

The subject invention is directed at an improved method for liquid-solid separation. More specifically, the subject invention is directed at a method for separating a solvent-oil-wax slurry into a substantially wax-free oil-solvent mixture and a waxy solid having a reduced solvent content.

It is well-known in the art to dewax wax containing hydrocarbon oils, particularly the lube oil fractions of petroleum oil, in order to remove at least a portion of the wax therefrom to obtain a dewaxed oil of reduced cloud and pour points. It also is well-known to deoil wax containing hydrocarbon oils, particularly slack wax from dewaxing operations, in order to remove at least a portion of the oil therefrom to obtain a crystalline wax product.

In current refinery practice, wax is separated from oils by various solvent dewaxing techniques. A summary of processes for oil wax separation is compiled in "Hydrocarbon Processing", September, 1978, pages 177-210, the disclosure of which is incorporated herein by reference. The wax containing hydrocarbon oil typically is mixed with solvent and cooled to crystallize the wax. The type, amount and distribution of solvent, as well as the specific hardware varies depending on the process. The resulting slurry of wax crystals in an oil-solvent mixture is separated by one of the methods to be described below.

The overall efficiency of the dewaxing operation depends to a large degree on the efficiency of the method of separating the wax crystals from the slurry. Several techniques have been developed for this separation:

1. Decantation
2. Filter pressing
3. Rotary filtration
4. Filtering centrifugation

Decantation and filter pressing, which are the oldest techniques, are not used commonly now due to their batch characteristics, which limit through-put.

One or more rotary filters may be used to separate the slurry into a substantially wax-free filtrate and a filter cake containing entrapped lube oil and about 3 to about 8 volumes of solvent per volume of wax. This method has several undesirable features. The filter cake produced may have an undesirably high entrapped oil and solvent content. This results in unacceptably high lube oil yield losses and necessitates the use of large wax recovery distillation trains in which the wax molecules are separated from the solvent. When the feed to the wax recovery train contains relatively high concentrations of solvent, the wax recovery unit must be operated at relatively low throughputs and a relatively high rate of energy input per unit of lube oil product.

Similarly, one or more rotary filters in series have been used to separate a slurry comprising a slack wax feed into a substantially wax-free filtrate and a refined wax filter cake containing entrapped lube oil and about 3 to about 6 volumes of solvent per volume of wax. This method is not preferred for producing a refined wax product, since it may be necessary to use an excessive amount of solvent to produce a wax having a sufficiently high melting point. Although it may be possible ultimately to recover most of the solvent added, the solvent recovery units must be operated at relatively low throughputs and relatively high rates of energy input per unit of refined wax product. In addition, since significant improvements previously have been made in increasing the throughput from rotary drum filters, the wax recovery operation frequently may be the rate limiting operation for the entire lube oil production unit and/or the solvent recovery operation may be the rate limiting operation for the entire refined wax production unit. Furthermore, rotary drum filters must be shutdown for washing with hot solvent at 4-10 hour intervals. Considerable energy is required to re-cool the filters to steady-state process conditions.

Various designs of centrifuges have been proposed for oil-wax separation. Apart from the batch centrifuges, which currently are not used for wax recovery, the most common design is the filtering centrifuge. Filtering centrifuges are known to be used only in systems where the density of the solvent is higher than that of the wax processed, such as where chlorinated solvent systems are used. In general, the operation of filtering centrifuges also is intermittent.

U.S. Patent No. 2,772,210 describes a solvent dewaxing process in which the large wax crystals are separated from the smaller, finer wax crystals present in a hydrocarbon oil by settling when the gravity differential between the wax and solvent oil phases is at least 0.05. When the gravity differential is less, the smaller crystals may be separated from the larger crystals by several methods including centrifugation. The solution including larger crystals, is then passed through the filter. The patent also discloses at column 2 that conventionally wax was separated from oil and solvent by filtration or centrifugation.

U.S. Patent Nos. 1,963,498; 1,999,468; 2,180,070; and 2,279,937, disclose the combination of filtration and centrifugation either to separate processing additives and wax from lube oil or amorphous wax from crystalline wax in a lube oil stream.

U.S. patent No. 3,006,839 discloses that a centrifuge may be used for wax deoiling, while U.S. Patent No. 1,989,028 discloses the use of a centrifuge followed by a filter for oil dewaxing.

U.S. Patent No. 2,723,941 discloses the use of two rotary or other filtration zones in series for wax deoiling.

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U.S. Patent No. 1,939,946 describes the use of a filtration zone for separating oil from wax. The filtrate from the filtration zone is passed to a centrifugation zone for further separation.

While the use of centrifuges either alone or in combination with filters may be known, the most common means for wax separation from solvent dewaxing slurries uses one or more filters.

5 Accordingly, to improve the dewaxing process efficiency, it is desirable to provide a process in which the wax separated from a lube oil-solvent-wax slurry contains reduced amounts of entrapped lube oil and solvent while not adversely affecting the cloud and pour points of the lube oil by wax entrainment into the lube oil product.

10 Similarly, to improve the slack wax process efficiency, it is desirable to provide a process in which the wax separated from a wax-lube oil-solvent slurry contains reduced amounts of entrapped lube oil and solvent.

It also is desirable to provide a process in which the solvent consumption is reduced to produce a refined wax product of predetermined purity.

15 It is also desirable to provide a process which is reliable and may be installed in existing facilities without extensive modifications.

It is also desirable to provide a process in which a centrifugation zone can be utilized for processing non-chlorinated solvents.

20 It has been found that the unique combination of a filtration zone followed by a centrifugation zone for processing a wax slurry produces a higher dewaxed oil yield and lower liquids content in the wax cakes than heretofore possible.

It has been found that the unique combination of a filtration zone followed by a centrifugation zone for processing a slack wax slurry may produce the combination of a higher deoiled wax yield and a wax cake having a lower liquids content than heretofore possible.

25 Summary of the invention

The subject invention is directed at an improved process for separating wax crystals from a hydrocarbon oil-solvent-wax slurry which comprises: (A) passing the slurry through a filtration zone to separate the slurry into a substantially wax-free filtrate and a filter cake having entrapped hydrocarbon oil and solvent; and, (B) passing filter cake from the filtration zone to a centrifugation zone wherein at least a portion of the entrapped hydrocarbon oil and solvent is removed from the filter cake.

30 In a preferred method the solvent utilized is less dense than the filter cake. Where a non-autorefrigerative solvent is used, the hydrocarbon feed-solvent mixture to the filter is chilled to a temperature between about -10°C to about -20°C to crystallize the wax and form a slurry. A solvent, such as for example MEK/MIBK, MEK/Toluene, or similar solvents, preferably is added in an amount ranging between about 3 and about 8 volumes per volume of feed to improve the feed viscosity and to assist in the separation of the hydrocarbon oil from the wax. When an autorefrigerative solvent such as propane, butane, or propylene alone or in admixture with one or more ketones, such as acetone, methyl ethyl ketone or methyl isobutyl ketone, is added, preferably in an amount ranging between about 1 volume per volume of feed and 3 volumes per volume of feed, the feed-solvent mixture is chilled to a temperature of about -20°C to about -40°C to crystallize the wax and form a slurry. Irrespective of the solvent used, the filtration zone preferably comprises one or more rotary drum filters, while the centrifugation zone preferably comprises a sedimenting type centrifuge, such as a scroll decanter centrifuge. At least a portion of the liquid separated in the centrifugation zone preferably is recycled to the filtration zone.

45 The present invention also is directed at a method for producing a refined wax product from a wax-hydrocarbon oil-solvent first slurry which comprises:

A. passing the first slurry through a filtration zone to separate the slurry into a substantially wax-free filtrate and a filter cake having entrapped hydrocarbon oil and solvent therein;

50 B. adding additional solvent to the filter cake from the filtration zone to produce a second slurry; and C. passing the second slurry to a centrifugation zone wherein hydrocarbon oil and solvent are separated from the wax.

55 In a preferred process, the feed comprises a slack wax from a dewaxing process, the slack wax having between about 3 and about 20 wt. % oil. The filtration zone preferably comprises a rotary drum filter and the centrifugation zone preferably comprises a sedimentation type centrifuge. Among the preferred sedimentation type centrifuges are the scroll-decanter, vertical decanter, and tubular centrifuges, with the most preferred being the scroll decanter centrifuge.

60 In a preferred method of operation, the first slurry enters the filtration zone at a temperature ranging between about -20°C to about $+30^{\circ}\text{C}$, preferably between about 0°C and about $+30^{\circ}\text{C}$, and the second slurry enters the centrifugation zone at a temperature ranging between about -20°C and about $+30^{\circ}\text{C}$, preferably between about 0°C and about $+30^{\circ}\text{C}$.

65 The solvent added to the first slurry and the second slurry preferably is the same and preferably is selected from the group of solvents consisting of methyl ethyl ketone, methyl isobutyl ketone, aromatic hydrocarbons, aliphatic hydrocarbons and mixtures thereof. The liquid, or centrate, separated by the centrifugation zone preferably is recycled directly to the filtration zone.

Description of the drawings

Figure 1 is a simplified flow drawing of one method for practicing the present invention.

Figure 2 is a simplified partial sectional view of a scroll decanter centrifuge useful for practicing the subject invention.

5 Figure 3 shows the four possible combinations of a filtration zone and a centrifugation zone for separating wax from a wax slurry and/or for deoiling a slack wax.

Detailed description of the invention

Referring to Figure 1, a simplified process flow diagram is shown. In this diagram, valves, piping, instrumentation and equipment not necessary for an understanding of the subject invention have been omitted for clarity. This feed is shown entering filtration zone 10 through line 12. When the feed is to be dewaxed, feed frequently comprises distillate from a vacuum pipe-still or deasphalted oil which may have been extracted and hydrotreated. The feed typically is chilled to a temperature ranging between about 0°C and about -40°C, preferably about -10 to about -20°C, by various means well-known in the art, such as indirect chilling utilizing a scraped surface heat exchanger, or by direct chilling. A solvent preferably is added to the feed to facilitate the hydrocarbon oil-wax separation. In direct chilling, a chilled non-volatile solvent, such as MEK/MIBK or MEK/Toluene or similar solvents is added to cool by intermixing, or a liquefied solvent, such as liquid propane, is injected and cools by vaporization. The solvent selected and the amount used are a function of several factors including the feed characteristics, and the desired product cloud and pour points. In addition to cooling the feed, solvents typically are added to improve the viscosity of the slurry for separation.

Where a refined wax is to be produced, the feed frequently comprises a slack wax from a dewaxing operation which is heated to a temperature between about -20°C and about +30°C, preferably between about 0°C and about +30°C, depending on the desired melting point of the refined wax product to facilitate the removal of low melting point wax from the refined wax product. Since the slack wax feed typically is reslurried by the addition of solvent to facilitate the separation, frequently the slack wax feed temperature is adjusted by regulation of the quantity and temperature of the solvent added. The temperature of the feed also may be adjusted by other means well known in the art, such as by indirect heat transfer. The solvent selected and the amount added are a function of several factors, including the slack wax feed characteristics and the desired crystalline melting point of the wax product. Preferred solvents are methyl ethyl ketone (MEK); methyl isobutyl ketone (MIBK); aromatic hydrocarbons, such as toluene or benzene; aliphatic, liquefied normally gaseous hydrocarbons, such as propane, butane, and butylene; and mixtures of these solvents.

Filtration zone 10 may comprise any type filter which is effective for liquid-solid separation. In the separation of a slurry of liquid hydrocarbon oil from solid wax crystals, a rotary drum filter previously has been found to be particularly effective, although other type filters, such as plate and frame filter presses, also may be used.

In oil-dewaxing, the filtrate from the filtration zone, which is essentially wax-free and contains about 55 to about 85 weight % solvent, is sent from filtration zone 10 to dewaxed oil recovery zone 30 through line 14. Dewaxed oil recovery zone 30 typically comprises a tower and associated equipment for the separation and recovery of solvent from the hydrocarbon oil. Recovered solvent is removed from recovery zone 30 through line 34 for recycle to the process, such as through lines 36, 38, 39 and 40, or to storage, while the dewaxed oil is removed from recovery zone 30 through line 32. The wax, having entrained hydrocarbon oil and solvent therein, forms a filter cake in zone 10 which is transported through line 22 to centrifugation zone 20 by conventional means, such as by a centrifugal pump (not shown). In the centrifugation zone 20 the filter cake, preferably at a temperature of about 0°C to about -20°C, is centrifuged; preferably with additional solvent repuddle, to remove additional quantities of solvent and oil from the wax. Typically, the filter cake entering centrifuge 20 contains about 3 to about 20 volumes of solvent per volume of wax, preferably about 8 to about 14 volumes of solvent per volume of wax, and about 15 to about 50 wt.% oil in the wax. The liquid removed from centrifugation zone 20 generally contains trace amounts of entrained wax. Typically, the liquid exiting centrifugation zone 20 comprises about 3 to about 7 wt.% hydrocarbon oil, about 97 to about 93 wt.% solvent, and less than 0.5 wt.% wax. Although the wax concentration in the liquid from centrifugation zone 20 may be unacceptably high for hydrocarbon lube oil product, this liquid may be recycled through lines 24, 39 and 40 where it is refiltered in filtration zone 10. The wax cake from centrifugation zone 20, preferably having a liquid-solid weight ratio of between about 0.8 and about 2.5, is transported from zone 20 to wax recovery zone 50 by conventional means, such as by melting and pumping. In wax recovery zone 50 the solvent is recovered from the wax by conventional distillation.

In refined wax production, the filtrate from the filtration zone, which commonly is referred to as foots oil solution, is essentially wax-free and contains about 50 to about 90 wt.% solvent. The foots oil solution is sent from filtration zone 10 to a foots oil separation zone 30 through line 14. Foots oil separation zone 30 typically comprises a distillation tower and associated equipment for the separation and recovery of solvent from the foots oil. Recovered solvent is removed from separation zone 30 through line 34 for recycle to the process, such as through lines 36, 38, 39 and 40, or to storage, while the foots oil is removed from separation zone 30 through line 32.

65 The wax, having entrained hydrocarbon oil and solvent therein, forms a filter cake in zone 10 which is

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transported from filtration zone 10 through line 22 to centrifugation zone 20 by conventional means, such as by a centrifugal pump (not shown). Prior to entering centrifugation zone 20, the filter cake is reslurried or "repuddled" by the addition of more solvent. In centrifugation zone 20 the slurry, maintained at a temperature of about -20°C to about $+30^{\circ}\text{C}$, preferably maintained at a temperature of about 0°C to about $+30^{\circ}\text{C}$, is centrifuged to remove additional quantities of solvent and oil from the wax. Typically, the slurry entering centrifuge 20 also contains about 3 to about 20 volumes of solvent per volume of wax, preferably about 8 to about 14 volumes of solvent per volume of wax, and about 0.5 to about 2.0 wt.% oil in the wax. Typically, the liquid, or centrate, exiting centrifugation zone 20 comprises about 2 to about 8 wt.% hydrocarbon oil, about 98 to about 92 wt.% solvent, and less than 0.5 wt.% wax. The centrate, which may contain minor quantities of wax, may be recycled through lines 24, 39 and 40 where it is refiltered in filtration zone 10. The wax cake from centrifugation zone 20, comprising refined hard wax and solvent, preferably having a liquid-to-solid ratio of between about 0.8 and about 2.5, is transported from zone 20 to solvent separation zone 50 by conventional means, such as by melting and pumping. In solvent separation zone 50 the solvent is recovered from the refined crystalline wax. In the process shown, the solvent passes through line 36 for use in reslurrying or repuddling the filter cake from filtration zone 10. The solvent also may pass through lines 38, 39 and 40 for slurring and washing the wax cake in filtration zone 10. The crystalline wax product is removed from separation zone 50 through line 52.

Where a chlorinated solvent is used for oil-dewaxing or refined wax production, the wax crystals typically are less dense than the solvent, while, when a non-chlorinated solvent is used, the wax crystals typically are more dense than the solvent. There are two general types of centrifuges which might be used in solvent-oil-wax systems, filtering centrifuges and sedimenting centrifuges. Centrifugal separations by filtering centrifuges, in which the liquid is filtered under centrifugal force, may be useful where a chlorinated solvent is used.

Where a non-chlorinated solvent is used, sedimenting centrifuges may be useful. The wax crystals, which are denser than the non-chlorinated solvent-oil mixture, such as ketone-oil or propane-oil, are subjected to a centrifugal field which separates the wax crystals by forcing the crystals radially outward. The crystals, which accumulate at the outer walls of the centrifuge, are removed by various mechanisms.

The type of centrifuge utilized may be important. Since the wax crystals may blind or plug filtration screens, sedimenting centrifuges, which do not utilize filters for separation, are preferred. Therefore, to avoid the problems associated with filtering type centrifuges, the present invention preferably is practiced utilizing a non-chlorinated solvent and a sedimenting type centrifuge, with the most preferred centrifuge being the scroll-decanter centrifuge.

Referring to Figure 2, a simplified schematic drawing is shown of the centrifuge utilized in testing the subject invention, a Sharples Model P660 scroll decanter centrifuge, 20 often also referred to as a solid-bowl centrifuge, 150 mm in diameter and 350 mm in length. A horizontal cylindrical rotor bowl 110, driven by a motor and gear means, (not shown), contains helical screw conveyor 120, rotating in the same or opposite direction but at a different speed, which is affixed to hollow shaft 130. Feed is introduced through shaft 130 and discharged into bowl 110 through opening 122, typically located near the end of the horizontal section of bowl 110. The slurry feed discharged is forced to travel around helical screw conveyor 120 by centrifugal force, causing the wax and liquid to separate. The wax deposits on the interior wall of bowl 110, while the liquid forms an inner ring, with the thickness of the ring determined by the height of overflow weir 140. As the liquid travels around helical screw conveyor 120, the liquid becomes clearer as it approaches overflow weir 140. Liquid, substantially free of entrained wax, passes over weir 140 for recycle to filtration means 10 as previously described. The wax layer is forced to travel in a direction opposite to that of the liquid by the difference in rotary speed between rotating bowl 110 and screw conveyor 120. The speed of wax discharged is directly proportional to the relative velocity of bowl 110 and screw conveyor 120. When bowl 110 and screw conveyor 120 are rotating in the same direction, bowl 110 typically rotates at a higher speed than screw conveyor 120. Thus, faster rotation of screw conveyor 120 in the same direction as the rotation of bowl 110 usually reduces the relative velocity between the bowl and the screw conveyor, thereby decreasing the rate of wax movement through centrifuge 20. The wax travels along conical beach section 112 for further drying prior to discharge through ports 150.

The ultimate hydrocarbon oil content in the wax discharged from centrifuge 20 is a function of several slurry characteristics and several variables in the operation of centrifuge 20. The slurry characteristics which affect the oil content of the wax discharged include slurry composition, temperature, viscosity and relative liquid-solid density. Variables in the operation of centrifuge 20 which affect the ultimate composition of the wax discharged include the feed rate, the speed of bowl 110 and screw conveyor 120, the height of overflow weir 140, as well as the length, diameter, feed point and beach angle, α , of the centrifuge.

During tests utilizing scroll decanter centrifuge 20 for lube oil dewaxing, the length, diameter, feed point and beach angle of centrifuge 20 were held constant. The specifications for the feeds used in the tests described hereinafter is presented in Table I. In these tests it was found that the scroll decanter centrifuge was able to operate continuously for a longer time than a conventional rotary drum filter. The scroll decanter centrifuge was operable even during feedstock changes.

TABLE I
Feed specifications


Grade	100N	150N	600N	1200N
API	33.1	31.2	27.2	25.0
Density at 15°C, g/cc	0.8592	0.8692	0.8916	0.8979
RI at 20°C	1.4756	1.4817	1.4920	1.4970
Viscosity at 37.8°C, cSt	18.81	27.54	135.43	326.70
Viscosity at 98.9°C, cSt	3.83	4.74	12.34	23.15
Pour point, °C	-19	-18	-11	-1
Cloud point, °C	-17	—	-21	—

20 Example I

Table II illustrates the superiority of the use of a filtration zone followed by a centrifugation zone for separating wax and hydrocarbon oil at high through-put rates for 150N distillate feedstock as compared with other filter-centrifuge combinations for dewaxing oil. In this table all possible combinations of centrifugation and filtration zones shown in Figure 3 were utilized. It may be seen that the use of a filtration zone and a centrifugation zone produced a wax having a lower oil content than either two filtration zones in series or two centrifugation zones in series. Reducing the oil content in the wax increases the dewaxed oil yield. Since wax recovery typically is accomplished by distillation, an energy intensive process, the subject invention results, in addition to yield gain, in energy savings over the present two stage filtration zone systems. Moreover, the present invention permits an increase in lube oil production where wax recovery is the production limiting operation. It also should be noted that the second stage centrifugation zones exhibited a much higher capacity than the first stage centrifugation zones. The first stage centrifugation zones had to be operated at a reduced through-put to produce a relatively wax-free filtrate. However, even if the first stage centrifugation zones were operated at reduced through-put, the entrained wax still may exceed product specifications. By comparison the second stage centrifugation zones could be operated at much higher through-puts, since the separated liquid was recycled to the first stage thereby eliminating the requirement that the separated liquid from the second stage centrifugation zones be wax-free.

TABLE II
Comparison of possible centrifugation zone—filtration zone combinations for lube oil de-waxing

Combination		Liquid entrained in wax cake*		Wt.% oil in wax		Wt.% wax in centrifuge liquid		Centrifuge capacity** (m ³ /h)	
1st Stage	2nd Stage	1st Stage	2nd Stage	1st stage	from 2nd stage	1st stage	2nd stage	1st stage	2nd stage
a. Filter	Filter	5.0	3.6	50	9	—	—	—	—
b. Filter	Centrifuge	5.0	1.8	50	4.7	—	<0.5	—	0.3
c. Centrifuge	Filter	3.8	2.2	50	4.7	<0.1	—	0.18	—
d. Centrifuge	Centrifuge	3.8	2.2	50	4.7	<0.1	<0.5	0.18	0.3

*Weight ratio of entrained liquid to solid wax in cake.

**Based on a 6" Sharples scroll decanter centrifuge.

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Example II

This Example illustrates in Table III, for each feedstock used, that a second stage centrifugation zone produced a wax product having a reduced oil content and also a reduced total liquid (solvent plus oil) content as compared to a second stage filtration zone for all feedstocks tested. Use of the second stage centrifugation zone produced a wax having at least 50% less liquid and a significantly lower oil content in the wax as compared to the wax produced using a second stage filtration zone.

TABLE III

Comparison of wax product characteristics for second stage filtration zone and second stage centrifugation zone

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Feed grade	Slurry to 2nd stage		Wax product from second stage filter		Wax product from second stage centrifuge	
	Dilution*	Wt.% oil in wax	Wt.% liquid in wax cake	Wt.% oil in wax	Wt.% liquid in wax cake	Wt.% oil in wax
100N	6.1	42	3.0	8	1.0	5.6
150N	7.7	35	3.6	9	1.2	4.7
600N	7.0	46	4.5	17	2.2	14.0
1200N	8.8	48	3.2	27	1.3	12.8

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*Wt solvent/wt (wax+oil).

Example III

As indicated below, the liquid contained in the wax can be reduced by increasing the differential scroll speed, since this increases the residence time of the wax in the centrifugation zone. A 150 N wax slurry from a first stage filtration zone had an oil content of approximately 40 wt.% at -12°C. This wax passed to a centrifugation zone maintained at constant operating conditions except for differential scroll velocity. The data is presented in Table IV below.

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TABLE IV

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Increasing the height of overflow weir 140 increases the centrifugation zone capacity for a given liquid content in the wax cake as indicated in Table V below for a 150 N feedstock where the desired liquid content in the wax cake was 2.2 wt.%.

TABLE V

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Weir height (cm)	Maximum capacity (m ³ /hr)
1.1	0.24
1.3	0.32
1.5	0.44

In the comparative tests and example described below a 600 Neutral slack wax feed was utilized for refined wax production. The entering slack wax feed contained 20 wt.% residual dewaxed oil. Several key properties of the feed from which the slack wax was produced were as follows:

	Density °API	28.3
	Density at 15°C, g/cc	0.885
5	RI at 20°C	1.4630
	Viscosity at 37.8°C, cSt	110
	Viscosity at 98.9°C, cSt	12
10	Dry wax at -12°C, wt. %	17
	Cloud point, °C	48

15 In the example and comparative tests described hereinafter, all possible combinations of filtration and/or centrifugation zones were utilized for refined wax manufacture. Simplified schematic flow diagrams of these processes are shown in Figure 3.

Example IV

20 The slack wax feed from a dewaxing operation was warmed up to a temperature of 25°C following dewaxing. Approximately 3 to 3.5 volumes of 40/60 v/v methyl ethyl ketone/methyl isobutyl ketone solvent was added to the slack wax feed to dissolve low melting wax and form a slurry. The slurry was passed through a filtration zone, comprising a rotary drum filter maintained at a temperature of 25°C. The wax cake exiting from the rotary drum filter was reslurried with about 4 volumes of 40/60 v/v MEK/MIBK solvent. This
25 second slurry was passed through a centrifugation zone comprising a Sharples model P850 vertical scroll decanter centrifuge at a feed rate of about 2 liters/min. Several key properties of the wax cake, the filtrate, the product wax and the solvent addition rate are presented in Table IV.

Comparative tests also were conducted in which other combinations of a filtration zone and centrifugation zone were utilized at substantially the same through-put rates as in Example IV. The results
30 also are presented in Table IV.

From a review of Table IV, it can be seen that the combination of a filtration zone followed by a centrifugation zone for refined wax production had the following advantages over other filtration zone and/or centrifugation zone combinations:

- A. The filtration zone-centrifugation zone combination required less solvent than a multi-stage
35 filtration system. This reduced the amount of solvent which subsequently had to be recovered;
- B. The filtration zone-centrifugation zone combination had a lower loss of refined wax to the foots oil stream than the multi-stage centrifugation system at comparable solvent addition rates; and
- C. The filtration zone-centrifugation zone combination had a lower refined wax loss than the centrifugation zone-filtration zone combination.

40 While the subject invention has been shown to be effective for lube oil dewaxing and for refined wax production utilizing a scroll decanter, or solid-bowl centrifuge, other types of sedimenting centrifuges, such as vertical decanting and tubular centrifuges, also may prove effective depending upon the range of oil-wax slurries to be treated, the feed rates and the desired final product characteristics.

45 Notes

"RI" is an abbreviation for refractive index.

RPM is an abbreviation for revolutions per minute.

Dimensions given in " (inch) are converted to cm by multiplying by 2.54.

TABLE IV

Comparison of possible filtration zone-centrifugation zone combinations for refined wax processing
Feedstock: 600 neutral slack wax deoiling temperature 25°C

Combination		Liquid entrained in wax cake*		Wt.% wax exiting in 1st stage filtrate or centrate	Wt.% oil in wax cake exiting 2nd stage	Total solvent on dewaxed feed**
1st Stage	2nd Stage	Exiting 1st stage	Exiting 2nd stage			
Filter	Filter	4.5	3.6	<0.1	1.3	1.4
Filter	Centrifuge	4.5	1.7	<0.1	1.3	1.2 est.
Centrifuge	Filter	2.2	3.6	0.2	1.3	1.2 est.
Centrifuge	Centrifuge	2.2	1.7	-0.2	1.3	1.1

*Weight ratio of entrained liquid to solid wax in cake.

20 **Volume/volume ratio.

Claims

- 25 1. A process for separating wax from a hydrocarbon oil-solvent-wax slurry which is characterized by:
 - (a) passing the slurry (12) through a filtration zone (10) to separate the slurry into a substantially wax-free filtrate (14) and a filter cake having entrapped hydrocarbon oil and solvent therein; and
 - (b) passing (via 22) filter cake from the filtration zone (10) to a centrifugation zone (20) wherein at least a portion of the entrapped hydrocarbon oil and solvent is removed from the filter cake.
- 30 2. The process of claim 1 wherein at least a portion of the hydrocarbon oil and solvent separated from the filter cake in the centrifugation zone (20) is returned (via 24) directly to the filtration zone (10).
3. The process of claim 1 or claim 2 wherein additional solvent is added to the filter cake prior to the filter cake entering the centrifugation zone.
4. The process of any one of claims 1 to 3 further characterized by additional solvent being added (via 36) to the filter cake to reslurry the filter cake prior to the resulting slurry entering into the centrifugation zone (20).
- 35 5. The process of any one of claims 1 to 4 further characterized by the temperature of the slurry in the filtration zone (10) being maintained between about -40°C and about +30°C.
6. The process of any one of claims 1 to 5 further characterized by the temperature of the slurry and/or filter cake entering the centrifugation zone (20) being maintained between about -20°C and about +30°C.
- 40 7. The process of any one of claims 1 to 6 further characterized by the slurry and/or filter cake passed to the centrifugation zone (20) containing from about 3 to 20 volumes of solvent per volume of wax.
8. The process of any one of claims 1 to 7 further characterized by the solvent being less dense than the filter cake.
9. The process of any one of claims 1 to 8 further characterized by the centrifugation zone (20) comprising a sedimentation-type centrifuge.
- 45 10. The process of any one of claims 1 to 9 wherein the sedimentation-type centrifuge comprises a scroll-decanter centrifuge.

50 Patentansprüche

1. Verfahren zur Abtrennung von Paraffinen (Wachs) aus einer Kohlenwasserstofföl-Lösungsmittel-Paraffin-Aufschlammung, dadurch gekennzeichnet, daß
 - 55 (a) die Aufschlammung (12) durch eine Filtrierzone (10) geleitet wird, um die Aufschlammung in ein im wesentlichen paraffinfreies Filtrat (14) und einen Filterkuchen mit eingeschlossenem Kohlenwasserstofföl und Lösungsmittel aufzutrennen, und
 - (b) der Filterkuchen (über 22) von der Filtrierzone (10) zu einer Zentrifugierzone (20) geleitet wird, in der mindestens ein Teil des eingeschlossenen Kohlenwasserstofföls und Lösungsmittels aus dem Filterkuchen entfernt wird.
- 60 2. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß mindestens ein Teil des Kohlenwasserstofföls und Lösungsmittels, das in der Zentrifugierzone (20) vom Filterkuchen abgetrennt worden ist, direkt (über 24) zu der Filtrierzone (10) zurückgeführt wird.
3. Verfahren nach Anspruch 1 oder 2, dadurch gekennzeichnet, daß dem Filterkuchen vor dem Eintritt in die Zentrifugierzone zusätzliches Lösungsmittel zugesetzt wird.
- 65 4. Verfahren nach einem der Ansprüche 1 bis 3, dadurch gekennzeichnet, daß zusätzliches

TABLE IV

Comparison of possible filtration zone-centrifugation zone combinations for refined wax processing
Feedstock: 600 neutral slack wax deoiling temperature 25°C

Combination		Liquid entrained in wax cake*		Wt.% wax exiting in 1st stage filtrate or centrate	Wt.% oil in wax cake exiting 2nd stage	Total solvent on dewaxed feed**
1st Stage	2nd Stage	Exiting 1st stage	Exiting 2nd stage			
Filter	Filter	4.5	3.6	<0.1	1.3	1.4
Filter	Centrifuge	4.5	1.7	<0.1	1.3	1.2 est.
Centrifuge	Filter	2.2	3.6	0.2	1.3	1.2 est.
Centrifuge	Centrifuge	2.2	1.7	0.2	1.3	1.1

*Weight ratio of entrained liquid to solid wax in cake.

20 **Volume/volume ratio.

Claims

- 25 1. A process for separating wax from a hydrocarbon oil-solvent-wax slurry which is characterized by:
 - (a) passing the slurry (12) through a filtration zone (10) to separate the slurry into a substantially wax-free filtrate (14) and a filter cake having entrapped hydrocarbon oil and solvent therein; and
 - (b) passing (via 22) filter cake from the filtration zone (10) to a centrifugation zone (20) wherein at least a portion of the entrapped hydrocarbon oil and solvent is removed from the filter cake.
- 30 2. The process of claim 1 wherein at least a portion of the hydrocarbon oil and solvent separated from the filter cake in the centrifugation zone (20) is returned (via 24) directly to the filtration zone (10).
3. The process of claim 1 or claim 2 wherein additional solvent is added to the filter cake prior to the filter cake entering the centrifugation zone.
4. The process of any one of claims 1 to 3 further characterized by additional solvent being added (via 36) to the filter cake to reslurry the filter cake prior to the resulting slurry entering into the centrifugation zone (20).
- 35 5. The process of any one of claims 1 to 4 further characterized by the temperature of the slurry in the filtration zone (10) being maintained between about -40°C and about +30°C.
6. The process of any one of claims 1 to 5 further characterized by the temperature of the slurry and/or filter cake entering the centrifugation zone (20) being maintained between about -20°C and about +30°C.
- 40 7. The process of any one of claims 1 to 6 further characterized by the slurry and/or filter cake passed to the centrifugation zone (20) containing from about 3 to 20 volumes of solvent per volume of wax.
8. The process of any one of claims 1 to 7 further characterized by the solvent being less dense than the filter cake.
9. The process of any one of claims 1 to 8 further characterized by the centrifugation zone (20) comprising a sedimentation-type centrifuge.
- 45 10. The process of any one of claims 1 to 9 wherein the sedimentation-type centrifuge comprises a scroll-decanter centrifuge.

50 Patentansprüche

1. Verfahren zur Abtrennung von Paraffinen (Wachs) aus einer Kohlenwasserstofföl-Lösungsmittel-Paraffin-Aufschlammung, dadurch gekennzeichnet, daß
 - 55 (a) die Aufschlammung (12) durch eine Filtrierzone (10) geleitet wird, um die Aufschlammung in ein im wesentlichen paraffinfreies Filtrat (14) und einen Filterkuchen mit eingeschlossenem Kohlenwasserstofföl und Lösungsmittel aufzutrennen, und
 - (b) der Filterkuchen (über 22) von der Filtrierzone (10) zu einer Zentrifugierzone (20) geleitet wird, in der mindestens ein Teil des eingeschlossenen Kohlenwasserstofföls und Lösungsmittels aus dem Filterkuchen entfernt wird.
- 60 2. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß mindestens ein Teil des Kohlenwasserstofföls und Lösungsmittels, das in der Zentrifugierzone (20) vom Filterkuchen abgetrennt worden ist, direkt (über 24) zu der Filtrierzone (10) zurückgeführt wird.
3. Verfahren nach Anspruch 1 oder 2, dadurch gekennzeichnet, daß dem Filterkuchen vor dem Eintritt in die Zentrifugierzone zusätzliches Lösungsmittel zugesetzt wird.
- 65 4. Verfahren nach einem der Ansprüche 1 bis 3, dadurch gekennzeichnet, daß zusätzliches



Lösungsmittel (über 36) zu dem Filterkuchen gegeben wird, um den Filterkuchen erneut aufzuschlämmen, bevor die resultierende Aufschlämmung in die Zentrifugierzone (20) eintritt.

5. Verfahren nach einem der Ansprüche 1 bis 4, dadurch gekennzeichnet, daß die Temperatur der Aufschlämmung in der Filtrierzone (10) zwischen etwa -40°C und etwa $+30^{\circ}\text{C}$ gehalten wird.

6. Verfahren nach einem der Ansprüche 1 bis 5, dadurch gekennzeichnet, daß die Temperatur der Aufschlämmung und/oder des Filterkuchens bei Eintritt in die Zentrifugierzone (20) zwischen etwa -20°C und etwa $+30^{\circ}\text{C}$ gehalten wird.

7. Verfahren nach einem der Ansprüche 1 bis 6, dadurch gekennzeichnet, daß die Aufschlämmung und/oder der Filterkuchen bei Weiterleitung zur Zentrifugierzone (20) etwa 3 bis 20 Volumina Lösungsmittel je Volumen Paraffin enthält.

8. Verfahren nach einem der Ansprüche 1 bis 7, dadurch gekennzeichnet, daß das Lösungsmittel eine geringere Dichte als der Filterkuchen aufweist.

9. Verfahren nach einem der Ansprüche 1 bis 8, dadurch gekennzeichnet, daß die Zentrifugierzone (20) eine Zentrifuge vom Sedimentationstyp umfaßt.

10. Verfahren nach einem der Ansprüche 1 bis 9, dadurch gekennzeichnet, daß die Zentrifuge vom Sedimentationstyp eine Schneckendekanterzentrifuge umfaßt.

Revendications

1. Procédé pour séparer la paraffine d'une suspension d'huile hydrocarbonée/solvant/paraffine, qui est caractérisée en ce que:

(a) on fait passer la suspension (12) par une zone (10) de filtration pour séparer la suspension en un filtrat (14) pratiquement dépourvu de paraffine et en un gâteau de filtre comportant de l'huile hydrocarbonée et du solvant emprisonnés;

(b) on fait passer (par 22) le gâteau de filtre provenant de la zone (10) de filtration vers une zone (20) de centrifugation dans laquelle au moins une partie de l'huile hydrocarbonée et du solvant emprisonnés est enlevée du gâteau de filtre.

2. Procédé selon la revendication 1, dans lequel au moins une partie de l'huile hydrocarbonée et du solvant séparés du gâteau de filtre dans la zone (20) de centrifugation est recyclée (par 24) directement vers la zone (10) de filtration.

3. Procédé selon la revendication 1 ou la revendication 2, dans lequel on ajoute un supplément de solvant au gâteau de filtre avant que ce gâteau de filtre n'entre dans la zone de centrifugation.

4. Procédé selon l'une quelconque des revendications 1 à 3, caractérisé en outre par l'addition d'un supplément de solvant (par 36) au gâteau de filtre pour remettre ce gâteau de filtre en suspension avant que la suspension résultante n'entre dans la zone (20) de centrifugation.

5. Procédé selon l'une quelconque des revendications 1 à 4, caractérisé en ce que l'on maintient entre environ -40°C et environ $+30^{\circ}\text{C}$ la température de la suspension dans la zone (10) de filtration.

6. Procédé selon l'une quelconque des revendications 1 à 5, caractérisé en outre en ce que l'on maintient entre environ -20°C et environ $+30^{\circ}\text{C}$ la température de la suspension et/ou du gâteau de filtre entrant dans la zone (20) de centrifugation.

7. Procédé selon l'une quelconque des revendications 1 à 6, caractérisé en outre en ce que la suspension et/ou le gâteau de filtre que l'on fait passer dans la zone (20) de centrifugation contient ou contiennent environ 3 à 20 volumes de solvant par volume de paraffine.

8. Procédé selon l'une quelconque des revendications 1 à 7, caractérisé en outre en ce que le solvant est moins dense que le gâteau de filtre.

9. Procédé selon l'une quelconque des revendications 1 à 8, caractérisé en outre en ce que la zone (20) de centrifugation est une centrifugeuse du type à sédimentation.

10. Procédé selon l'une quelconque des revendications 1 à 9, dans lequel la centrifugeuse du type à sédimentation comprend une centrifugeuse à dispositif cylindrique de décantation.

