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KETONE DEWAXING PROCESS

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This invention concerns an improved dewaxing process directed to the solvent dewaxing of petroleum oil fractions containing wax. In accordance with this invention particular petroleum fractions are diluted with a ketone type solvent, and are heated sufficiently to dissolve the wax present in the petroleum fraction. The mixture of the solvent and the petroleum fraction is then cooled at a critical rate while maintaining a critical amount of agitation to cause the formation of wax crystals which may be filtered readily from the petroleum oil fraction. The operation of this process in accordance with this invention is effective in providing greatly improved filter rates, and improved yields of dewaxed oil.

Since about 1930 one of the commercial processes for recovering wax from petroleum oils has been the "ketone dewaxing process." While other ketones may be employed in this process, it has been the general practice to employ methyl ethyl ketone (in admixture with aromatics), as the solvent, so that the process has generally been known as the methyl ethyl ketone, or "MEK dewaxing process." Commercial MEK dewaxing processes simply require the addition of a suitable quantity of the MEK solvent to the oil to be dewaxed so as to permit complete solution of all wax present in the oil when the mixture is heated. After the wax has been dissolved, upon cooling down the mixture of oil and ketone, the wax is precipitated and is removed from the oil by filtration. While, as indicated, the MEK process has been in commercial use for a great many years, certain deficiencies of this process have become apparent. For example, it has been found that with certain types of oil stocks extremely poor filter rates are obtained, presumably due to the formation of wax crystals which are difficult to separate from the oil. A concomitant of this difficulty is that the oil content of the wax is generally at a level which is undesirably high, and in turn results in poor yields of dewaxed oil. Consequently, it is the principal object of this invention to materially improve the filter rates, to improve dewaxed oil yields, and to decrease the oil content of the wax obtained in the ketone dewaxing process.

Throughout this disclosure, the solvent to be employed will be referred to as a ketone, or specifically as methyl ethyl ketone. However, it is to be understood that any ketone containing from 3 to 6 carbon atoms may be employed. Again, it is to be understood that the solvent consisting primarily of a ketone of this class may also include up to 50%, or more commonly, from 25 to 50% of an aromatic compound or compounds employed to favorably supplement the solvent action of the ketone. Benzene and toluene are examples of aromatic constituents which may

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and often are included with the lower molecular weight ketones such as methyl ethyl ketone to provide the dewaxing solvent.

In employing ketone type solvents of the nature described, the oil to be dewaxed is diluted with the solvent and is then heated to a sufficient temperature to dissolve all wax present in the oil. On cooling the oil-solvent-wax solution by use of heat exchanging chilling surfaces, the wax is precipitated from the solution and may be recovered. The use of chilling surfaces or the "external chilling" of the solution, is a characteristic of the ketone dewaxing process, clearly differentiating the process, and problems connected with the process, from other dewaxing operations such as "propane dewaxing" wherein "internal cooling" is used, provided by evaporation of the solvent.

As indicated, the process of this invention is of specific application to a particular type of oil feed stock. It has long been known that the type of wax which may be recovered from an oil is dependent upon the boiling range of the oil fraction. Thus, for example, the wax recovered from oil fractions boiling up to about 850° F. at atmospheric pressure consists almost entirely of paraffin, or crystalline type wax. Similarly, the wax recovered from oils boiling above about 950° F. at atmospheric pressure consists principally of micro crystalline waxes. In general no particular problems have been involved in the dewaxing of oil stocks containing either paraffin wax, or micro crystalline wax. However, the problems of poor filtration rate, poor dewaxed oil yield, etc., referred to above, have been found to be peculiar to oil stocks containing a mixture of paraffin and micro crystalline waxes. These stocks are petroleum oil fractions having a boiling range such that at least 10% (based on ASTM 10 mm. mercury distillation) boils between about 540° and 620° F. at 10 mm., or 850 to 950° F. equivalent temperatures at atmospheric pressure. The problems are particularly severe in the dewaxing of distillate stocks in which the major portion of the stock boils in this range; that is 850° to 950° F. at atmospheric pressure. It must be understood, therefore, that the process of this invention is of especial application to this particular type of stock, characterized by the inclusion of both paraffin and micro crystalline waxes and by inclusion of a substantial portion of oil boiling in the range of 850° to 950° F. at atmospheric pressure.

In accordance with this invention, it has been found that in the treatment of the specified oil feed stock, a critical combination of process variables exists controlling effective dewaxing operations. In particular, it has been found that a critical amount of solvent dilution should be employed, providing a total liquid to solid

weight ratio of between about 15 to 1 and 25 to 1 at the dewaxing temperature. With the feed stocks normally encountered in petroleum refining, a dilution ratio of about 2 to 4 volumes of solvent per volume of oil to be treated will meet this requirement. The chilling rate to be employed should be below about 5° F. per minute and preferably within the practical range of 2 to 5° F. per minute. Finally, the amount of agitation to be used during chilling, prior to filtration, is critically selected so that no wax is deposited on the cooling surfaces employed to chill the oil-solvent-wax solution.

In order to fully disclose the nature of this invention, and to bring out the advantages thereof, data will be given in the following examples showing the improved results which may be secured by regard to the critical process variables of dilution ratio, chilling rate, and agitation.

EXAMPLE I

In order to determine the effect of agitation and chilling rate in ketone dewaxing, a conventional ice cream freezer of three gallons capacity was employed. The freezer was modified by installation of a variable speed drive to permit variation in the amount of agitation, and was also modified by positioning a thermocouple in the agitator shaft to permit accurate determination of chilling rates. Springs were employed to hold the scraper blades against the sides of the freezer. In operating this freezer at a speed of 9 revolutions per minute, a close approximation to the agitation conventionally used in commercial ketone plants was obtained. Thus, in present ketone dewaxing plants, chilling is carried out in six inch pipes equipped with scrapers rotating at about 12 R. P. M. The 9 R. P. M. freezer operation comprised a 9 R. P. M. turning of the scraper in one direction, and a 9 R. P. M. turning of the paddle and container in the opposite direction so that the speed of the scraper relative to the container was 18 R. P. M., or somewhat greater than the speed of the plant scrapers. It should be noted that the degree of agitation provided by the scrapers in commercial plants, or in the freezer operated at 9 R. P. M. is substantially nil. Under these conditions, the rotation of the scrapers is sufficiently slow so that the liquid is virtually undisturbed by the scraper, so that no turbulence whatever is induced. Actually, the operation of the scrapers in commercial ketone plant chillers has not been intended to provide agitation, but rather has been intended solely to mechanically scrape wax from the chilling surfaces. Furthermore, flow through the six inch diameter chillers of commercial plants, is conventionally conducted in parallel, so as to further eliminate any possibility of turbulence induced by flow.

In order to provide positive agitation in laboratory investigations, the freezer described was further modified by substitution of a propeller agitator for the scraper and paddle agitator. For this purpose an air driven propeller stirrer was used having three blades with an angle of 30° from vertical for downward thrust, and having a diameter of 2.75 inches. As the propeller was operated at sufficiently high speeds to prevent deposition of wax on the wall of the freezer, when using the propeller it was unnecessary to employ the scraper to remove wax from the wall, so that the scraper and paddle could be dispensed with and the propeller substituted therefor.

In a first series of experiments, a lubricating oil distillate was subjected to varying degrees of agitation in a series of runs in which all other variables were maintained constant. The lubricating oil distillate had the following inspections:

Table I

Gravity, ° API	31.9
Flash, C. O. C., ° F	470
Viscosity, S. S. U., at 210° F	53.8
Pour, ° F	120
Dry wax content, weight per cent	[1] 19.0
Distillation at 10 mm. Hg:	
I. B. P	310
5	430
10	517
20	560
30	578
40	590
50	599
60	608
70	620

[1] For 20° F. pour dewaxed oil.

In each of the runs made the dilution ratio was maintained at 3.3 volumes, resulting in a total liquid to solid ratio at filtering temperature of 21 to 1. In all experiments the solvent employed consisted of 50% methyl ethyl ketone; 29% benzene and 21% toluene. Solvent was added in the conventional manner by diluting the total oil feed, prior to chilling, with the total amount of solvent employed. Sufficient cooling was provided through the wall of the container to provide a chilling rate of 2½° F. per minute. Crystallization of wax was conducted by first heating the oil and solvent to a temperature of 118° F., and then cooling to a temperature of 0° F. at the indicated chilling rate. The wax was filtered from the wax-oil-solvent slurry with a leaf type filter in a period of 30 seconds, after which any desired amount of wash solvent could be applied to the wax cake. The results of these runs are indicated in Table II below:

Table II

Agitator	Scraper and Paddle		Propeller		
Agitator Speed, R. P. M.	9	90	650	1,250	2,100
Filter Rate, Gal. Dewaxed Oil/hr./sq. ft.[1]	3.8	5.3	5.6	6.1	6.3
Dewaxed Oil Yield (no wash), Vol. Percent	33	56	63	66	69
Wax Oil Content (no wash), Wt. Percent	70	56	50	46	39
Wash for 20% Oil Content, v./v. Feed	>3.5	1.7	1.2	0.8	0.5

[1] Without wash.

It will be noted from Table II that a progressive and substantial improvement in the dewaxing operation was secured by successively increasing the degree of agitation. Thus, for example, in referring to the filter rate obtainable, reported in gallons of dewaxed oil per hour per square foot of filter, the filter rate was only 3.8 when an agitator speed of 9 R. P. M. was employed. As indicated, this method of operation closely corresponds to the conventional commercial operation presently employed. However, on increasing the rate of agitation to 90 R. P. M. employing the same scraper and paddle arrangement, the filter rate was increased to 5.3. Finally, by substituting a propeller for the scraper and paddle, and by employing propeller speeds of 650, 1200, and 2100 R. P. M., the filter rate was improved respectively to 5.6, 6.1 and 6.3. This same relationship exists in regard to the dewaxed oil yield obtained, since

the yield increased from 33 for the minimum agitation, up to 69 for the maximum agitation. Again, considering the factor of the oil content of the wax, it will be noted that the wax cake obtained by the conventional method in which little agitation is employed, was a cake having an oil content of 70 weight per cent. However, the oil content of wax cakes obtained when greater agitation was used during chilling was reduced to a figure of 39 weight per cent for an agitation of 2100 R. P. M., using the propeller. Finally, Table II indicates the amount of solvent wax required to reduce the oil content of the cake to an oil content of 20%. The wash employed, was of the same composition as the solvent and was reported as volumes of wash per volume of feed. In the case in which substantially no agitation was employed, as given by the column for 9 R. P. M. agitator speed employing a scraper and paddle, it was found that more than 3.5 volumes of wash per volume of feed were required to reduce the wax to an oil content of 20%. However, in the washing of the wax cake obtained when employing greater agitation, successively smaller quantities of wash were required, so that in the case of the wax cake obtained by agitation at 2100 R. P. M., only 0.5 volume of wash per volume of feed were required to reduce the wax to an oil content of 20%. These improvements result from the more favorable crystallization conditions provided, which allow the formation of relatively uniform, regularly shaped crystals, which form a dense and yet highly permeable, easily washed wax cake.

It is apparent from these data, therefore, that the factor of agitation during crystal formation in ketone dewaxing is extremely important. As indicated, it is possible to substantially double the filter rate, to double the dewaxed oil yield, to decrease the oil content of the wax cake to almost one-half, and to enormously reduce the wash required for a wax cake of low oil content. It is apparent from the data presented, that material advantages are obtained by progressively increasing the degree of agitation employed. Thus, as against the case in which a 9 R. P. M. agitation rate is maintained providing substantially no turbulence in the mixture, material advantages are secured in any case in which sufficient agitation is used to create substantial turbulence in the mixture during chilling. The amount of agitation which should be employed may, therefore, be said to be at least the amount of agitation required to turbulently mix the oil solvent mixture during chilling so as to maintain a uniformly mixed or homogeneous mixture. Homogeneity can readily be determined by noting whether or not wax crystallizes on the cooling surfaces provided. Thus, in the case in which the paddle and scraper was turned at 9 R. P. M. homogeneity or uniform mixture was not secured, and wax precipitated on the chilling surfaces, necessitating use of the scraper. However, when employing the propeller agitator at speeds above 650 R. P. M. homogeneity was secured, and no wax precipitated on the chiller even though no mechanical scraping was done. With regard to the extent of agitation to be employed in a ketone dewaxing operation, therefore, in accordance with this invention sufficient agitation is used so as to provide substantial turbulence to maintain the mixture as a homogeneous mixture and to impart sufficient motion at the externally chilled surfaces to prevent deposition of wax on these surfaces.

EXAMPLE II

Insofar as data of Example I related to an operation in which all of the solvent was added to the oil at the initiation of the chilling, further experiments were conducted in which a different method of solvent addition was employed. In particular a so-called "incremental method" of solvent addition was used in which the solvent was added in increments during the chilling operation. Thus, 0.3 volume of solvent was added at 130–140° F., 0.3 volume of solvent was added at 80° F., 0.7 volume of solvent was added at 40° F., and 2.0 volumes of solvent was added at 15° F. For comparative purposes the runs employing this method of solvent addition were supplemented by runs in which the conventional method of adding the total solvent at the beginning of the chilling was employed. The lubricating oil distillate employed in these tests, the chilling rate used, the filtering technique and all other variables were maintained as described in Example I.

In a first test, conventional solvent addition was carried out, or 3.3 volumes of solvent per volume of feed were added at the beginning of the chilling, at a temperature of 130 to 140° F. The 9 R. P. M. agitation was maintained during chilling. It was found that the filter rate obtained was 3.8 gallons of dewaxed oil per hour, per square foot. In a comparable test employing the incremental solvent addition method described above, and employing the same agitation rate of 9 R. P. M., it was found that the filter rate obtainable was substantially the same, or about 3.9. In accordance with this invention, agitation was used to maintain a homogeneous mixture during chilling by employing a propeller agitator operating at 2100 R. P. M. In the case of conventional solvent addition, it was found that a filter rate of 6.4 was obtained, while with incremental solvent addition a filter rate of 4.9 was obtained. In both runs at 2100 R. P. M., motion of the slurry at the chilled surfaces was sufficiently vigorous so that no appreciable buildup of wax occurred, even though no scraping was employed. It is apparent from these data that the material advantages of providing a high degree of agitation during chilling may be appreciated when either the conventional, or incremental method of solvent addition is employed. The complete data for the above-mentioned runs with incremental solvent addition and for an additional run conducted at 90 R. P. M. employing the scraper and paddle agitator are given in Table III.

Table III

EFFECT OF AGITATION DURING CHILLING; SOLVENT ADDITION METHOD—INCREMENTAL

Agitation Rate. R. P. M.	9 [1]	90 [1]	2,100 [2]
Filter Rate, gal. Dewaxed Oil/hr./ft.² [3]	3.9	4.2	4.9
Dewaxed Oil Yield (No Wash), Vol. percent	60	66	71.5
Wax Oil Content, Wt. percent	51	44	31
Wash Requirement for 20% Oil Content, v./v. Feed	1.2	0.7	0.2
Wax Cake Volume, v./v. Feed	1.2	0.9	0.6
Wax Cake Thickness, inches	0.26	0.19	0.15

[1] Ice cream freezer (double scraper rotating in opposite direction to that of can and paddle).
[2] Air driven, propeller stirrer in baffled system.
[3] 30 seconds' filtration time, no wash.

As established, therefore, the degree of agitation provided is a processing factor which must be critically maintained regardless of the method of solvent addition employed; or conversely, the objectives of this invention may be attained in

ketone dewaxing irrespective of the manner in which the solvent is added.

EXAMPLE III

While as indicated in accordance with this invention a high degree of agitation is maintained during chilling in ketone dewaxing, it is necessary for best results that this agitation should not be continued after the chilling has been completed. Thus, the benefits resulting from thorough agitation during chilling are in part offset if the agitation is continued after the filtering temperature has been attained. Thus, data were obtained employing the feed stock and process variables given in Example I, except that agitation was continued after chilling while maintaining the mixture at the filtering temperature. Referring to the data of Example I, it will be noted at 9 R. P. M. agitation, employing a paddle and scraper, a filter rate of 3.8 gallons of oil per hour per square foot is obtainable. This data was obtained when the agitation was maintained only during chilling and was discontinued as soon as the filtering temperature of 0° F. was reached. The filter rate, as indicated, may be increased to 5.3 by increasing the rate of agitation to 90 R. P. M. using the paddle and scraper, similarly discontinuing agitation when the filtering temperature has been attained. In a comparative run at 90 R. P. M., agitation was, however, continued for five to ten minutes at the filtering temperature. In this case it was found that the filter rate obtained was only 4.2 gallons of dewaxed oil per hour, per square foot. These data, therefore, show that in the practice of this invention, while thorough agitation during chilling is extremely beneficial, the continuation of agitation at the filtering temperature is disadvantageous and largely offsets the advantages obtained by agitation. This same principle is true at higher degrees of agitation, although as the degree of agitation increases, the drop in filter rate caused by agitation at the filtering temperature becomes somewhat less. In this connection, it may be noted that an additional advantage of maintaining vigorous agitation during chilling is that the resultant wax slurry is less sensitive to agitation occurring at the filtering temperature.

According to the data of this example, therefore, it is advantageous in the practice of this invention to discontinue vigorous agitation once the filtering temperature has been attained.

EXAMPLE IV

A series of runs were conducted in which varying chilling rates were employed while maintaining turbulent agitation as taught by this invention. In these experiments the lubricating oil was that identified in Example I; the solvent was that of Example I, and the dilution ratio was maintained as there stated. However, two different chilling rates were employed as indicated in Tables IV and V, below:

Table IV

Solvent Addition Method	Conventional		Incremental	
Chilling Rate, ° F./minute	2.3	6.7	2.7	5.0
Freezer Speed, R. P. M.	90	90	90	90
Filter Rate, Gal. Dewaxed Oil/hr./sq. ft.[1]	5.3	4.5	4.2	3.6
Dewaxed Oil Yield (No Wash), Vol. Percent	56	47	66	64
Wax Oil Content (No Wash), Wt. Percent	56	64	44	47
Wax Cake Volume, v./v. Feed	1.3	1.6	0.9	0.9

[1] Thirty seconds' filtration time.

Table V

Propeller Agitation, R. P. M.	2,100	
Solvent Addition Method	Conventional	
Chilling Rate, ° F./minute	2.5	9.6
Filter Rate, Gal. Dewaxed Oil/hr./sq. ft.[1]	6.3	4.1
Dewaxed Oil Yield (No Wash), Vol. Percent	69	61
Wax Oil Content (No Wash), Wt. Percent	39	52
Wash Required for 20% Oil Content Vol. of Wax, v./v. Feed	0.5	0.9

[1] Thirty seconds' filtration time.

It will be noted from Tables IV and V that material dewaxing improvements were obtained by employing the slower chilling rates. Thus, when the solvent was added according to the conventional method; that is, in bulk at the beginning of the chilling, material operational improvements were obtained at chilling rates of 2.3° F. per minute as against a chilling rate of 6.7° F. per minute (Table IV) or at 2.5° F. per minute rather than 9.6° F. per minute (Table V), at 90 R. P. M. and at 2100 R. P. M. agitation rate, respectively. Higher filter rates, higher yields of dewaxed oil, a wax cake having smaller volume and holding less oil was obtained when the lower chilling rates were employed. These same correlations existed when the solvent was added by the incremental addition method employing the comparative chilling rates of 2.7 and 5.0° F. per minute (Table IV). It is apparent from these data, therefore, that high chilling rates are to be avoided during the crystallization step of a ketone dewaxing process. Since chilling rates below about 2° F. per minute are considered impractical commercially, it appears that chilling rates of about 2 to 3° F. per minute are to be preferred, although rates of 2 to 5° F. per minute may be employed. As evidenced by the data, chilling rates greater than about 5° F. per minute materially reduce the filter rate obtainable, the yield of dewaxed oil, etc.

As established by the data of this example, therefore, not only is a high degree of agitation required for best results in ketone dewaxing but, furthermore, chilling rates should be carefully controlled so as not to exceed about 5° F. per minute, and preferably so as to provide chilling at the rate of about 2 to 3° F. per minute.

EXAMPLE V

A series of experiments were conducted to determine the relation of improvement secured by agitation to the nature of stock dewaxed. The effects of increased agitation in the ketone dewaxing of a variety of petroleum fractions are summarized in Table VI.

Table VI

Feed Stock	A	B	C	D	E	F
Wax Content, Wt. Percent	49	21	6	18	19	22
10 mm. ASTM Distillation:						
5% off at ° F.	429	403	418	420	430	568
95% off at ° F.	592	579	619			
Percent off at 620° F.				84	70	9
Vol. percent in Range of 540-620° F. at 10 mm. Hg (850-950° F. at atm. pressure)	18	15	23	58	55	9
Effects of Increased Agitation During Chilling[1]:						
Increase in Filter Rate, Percent	0	6	5	68	66	[2] −34
Decrease in Wash Required to reach 20% Oil Content Wax, Percent	0	64	80	73	86	(2)

[1] 2,100 R. P. M. propeller speed vs. 9 R. P. M. paddle and scrapers.
[2] Disadvantage for increased agitation.

It will be noted that stocks D and E, which include 58 and 55%, respectively, of material distilling within the critical range of 540 to 620° F. at 10 mm. of mercury pressure, show the most marked improvement with the use of vigorous agitation during chilling. Lower boiling stocks B and C, which contain 15 and 23% of material distilling within the critical range, show only a slight increase in filter rate but a marked improvement in terms of wash requirement. Lower boiling stock A, which contains 18% of material distilling within the critical range, appears unaffected by increased agitation, apparently by virtue of the fact that this particular stock contained substantially no microcrystalline type wax. At the other extreme, stock F, which is a high boiling, residual type stock containing only 9% boiling below 620° F. at 10 mm. of mercury pressure, shows impaired results (lower filter rate, increase in wash requirement) with the use of more viborous agitation during chilling. This invention does not apply, therefore, to low boiling stocks containing substantially no microcrystalline type wax and having less than 10% of material distilling above 540° F. at 10 mm. of mercury pressure, or to high boiling stocks containing largely microcrystalline type wax and having less than 10% distilling below 620° F. at 10 mm. of mercury pressure.

As heretofore described, and as brought out in the examples given, the process of this invention is a particular ketone dewaxing process of application to a particular type of feed stock. Thus, it has been shown that in treating a distillate fraction of which a substantial portion boils in the range of about 540–620° F. at 10 mm. of mercury absolute pressure (850–950° F. at atmospheric pressure) material dewaxing improvements are secured according to the process described. In the processing of such stocks, a chilling rate of about 2 to 5° F. per minute is maintained, a dilution ratio of about 2 to 4 volumes per volume of feed is maintained, and a critical amount of agitation is employed. Sufficient agitation or turbulence is maintained during chilling to provide a thoroughly mixed, homogeneous mixture of crystallized wax, solvent and oil. It is preferred that after the chilling, the agitation be substantially discontinued.

The temperature to which the mixture of solvent and oil is heated prior to chilling may best be defined as the temperature at which all wax present will be dissolved. Dependent on the particular oil stock treated, this temperature will lie in the range of about 100° to 160° F. or more narrowly will generally fall between 120° F. to 150° F. The temperature to which the mixture is chilled to secure dewaxing is commonly between 0 to +25° F. but may be any temperature in the range of −10° F. to +40° or 50° F. dependent in part on whether the objective is to recover wax or to secure a low pour point oil.

The manner in which agitation during chilling is secured is not a part of this invention. Thus any desired means may be used to maintain a homogeneous mixture of wax, solvent and oil during crystallization. In the case of existing equipment, the conventional scraper operated chillers may be modified, for example, by providing a clearance space between the scrapers and the chilled surface and by operating the scrapers at a much higher turning rate. Again, this same result may be secured by removing the scrapers and replacing them with propeller agitators, or other agitating means to maintain a uniform mixture. As still another possibility, the solvent-oil mixture may be subjected to shear type dispersing apparatus, as for example a homogenizer during the wax crystallization. Again, it is contemplated that ultrasonic vibrations may be employed to secure the degree of mixing required.

What is claimed is:

1. A process for dewaxing a waxy petroleum distillate, a substantial portion of which boils in the range of 850° F. to 950° F. at atmospheric pressure, and characterized by inclusion of both paraffin and microcrystalline type waxes, comprising the steps of adding to said distillate a solvent comprising principally a ketone having from 3 to 6 carbon atoms, and heating said mixture of solvent and distillate to a temperature sufficient to dissolve wax present in the distillate, and thereafter cooling said mixture of solvent and distillate at a rate of 2° to 5° F. per minute by heat exchange through chilling surfaces, while maintaining sufficient agitation during chilling and wax crystallization to maintain a turbulent, uniformly mixed slurry of crystallized wax, solvent, and distillate characterized by the absence of crystallized wax on the said chilling surfaces.

2. The process defined by claim 1 in which not less than 10% of the said distillate boils above 850° F. and more than 10% of the said distillate boils below 950° F.

3. The process defined by claim 1 in which the said solvent includes up to 50% of an aromatic hydrocarbon.

4. The process defined by claim 1 in which the said solvent includes up to 50% of an aromatic hydrocarbon selected from the class consisting of benzene and toluene.

5. The process defined by claim 1 in which the said ketone is methyl ethyl ketone.

6. The process defined by claim 1 in which about 25% of the said distillate distils in the range of 850° F. to 950° F. at atmospheric pressures.

7. The process defined by claim 1 in which about 50% of the said distillate distils in the range of 850° F. to 950° F. at atmospheric pressures.

8. The process defined by claim 1 in which a major portion of the said distillate distils in the range of 850° F. to 950° F. at atmospheric pressures.

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References Cited in the file of this patent

UNITED STATES PATENTS

Number	Name	Date
2,229,658	Jenkins	Jan. 28, 1941
2,319,381	Wickham et al.	May 18, 1943
2,397,868	Jenkins	Apr. 2, 1946
2,410,483	Dons et al.	Nov. 5, 1946
2,478,863	Davis	Aug. 9, 1949
2,578,192	Mair	Dec. 11, 1951