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SOLVENT DEWAXING PROCESS

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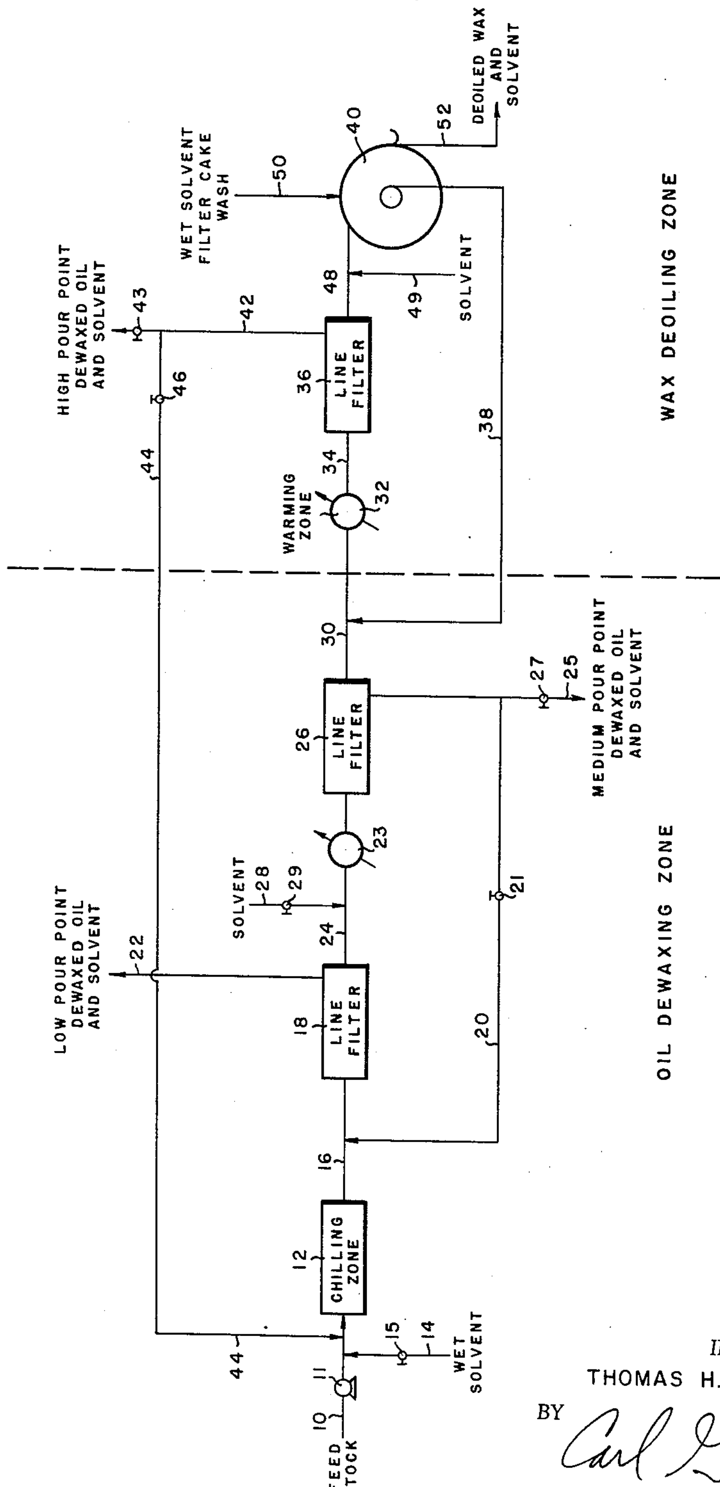

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SOLVENT DEWAXING PROCESS

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This invention relates to a method for the solvent dewaxing of petroleum hydrocarbon lubricating oil fractions. More particularly, this invention relates to an improved method for the solvent dewaxing of petroleum hydrocarbon lubricating oil fractions wherein an enhanced rate of recovery of dewaxed oil is obtained. Still further, this invention relates to an improved combination process for the simultaneous dewaxing of oil and deoiling of wax.

Paraffinic petroleum hydrocarbon fractions which are suitable for use as base materials in the compounding of motor oils will normally contain a significant amount of wax which must be removed if a satisfactory motor oil is to be obtained. The wax is normally removed from such feed stocks in a continuous process by fractional crystallization of the wax from a solution of the feed stock in a dewaxing solvent. In order to remove the wax economically, it is desirable to provide for as high a ratio as possible of recovered dewaxed oil to dewaxing solvent. This is necessary in order to provide for a minimized plant construction cost, plant operation cost, and plant maintenance cost.

In addition, it is frequently desirable to treat the wax resulting from a dewaxing operation in order to remove oil impurities therefrom to thereby provide a purified wax which is useful in commerce.

It has been discovered in accordance with the present invention that the amount of dewaxed oil obtained in a continuous process for a given solvent inventory may be substantially increased by a continuous process wherein a solution of a waxy feed stock in dewaxing solvent is chilled to an extent sufficient to form a pumpable slurry of about 5 to 15 volume percent of wax crystals in a mother liquor comprising the solvent-oil solution, wherein the slurry is turbulently isothermally filtered to an extent sufficient to remove from about 20 to 60 volume percent of the mother liquor, wherein the concentrated slurry is diluted with an additional quantity of solvent sufficient to provide a second slurry having about the same concentration of wax crystals as the original slurry and subjecting the second slurry to a second turbulant isothermal filtration step to obtain a recycled filtrate fraction and a concentrated wax fraction, the recycled filtrate fraction being admixed with the initial wax slurry prior to the initial turbulent isothermal filtration step.

In accordance with one aspect of the present invention, further advantages are obtained in that a flexible process is provided wherein several grades of dewaxed oil of controlled pour point may be obtained or wherein a single grade of dewaxed oil is obtained having a pour point controlled by the selective rejection of all or a portion of at least one recycle fraction.

The charge stocks of the present invention are lubricating oil petroleum hydrocarbon distillate fractions boiling within the range of about 420° to about 800° F. at a pressure of 10 mm. of mercury and containing from about 10 to 40 weight percent of paraffinic wax components.

The dewaxing solvent to be employed is preferably a mixture of a precipitating type solvent with a diluent type solvent. Thus, the precipitating type component may be an aromatic compound. Thus, ketones such as acetone, methylethylketone, methyl-n-propyl ketone, diethylketone, etc. and mixtures thereof may be employed in connection with an aromatic diluent type solvent such as benzene, toluene, xylenes, etc. and mixtures thereof. As another example, a precipitating type solvent such as ethylene dichloride may be employed in conjunction with a diluent type component such as benzene, chloroform, carbon tetrachloride, etc. and mixtures thereof. As a further example, the precipitating component may be furfural and the diluent component may be an aromatic compound such as benzene.

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In general, the dewaxing solvent will contain a major amount of the precipitating type component and a minor amount of the diluent type component. As is known to those skilled in the art, the ratio of precipitant type component to diluent type component will be dependent, in large measure, upon the dewaxing temperature to be employed, the characteristics of the charge stock to be dewaxed and the desired product quality. Thus, a workman skilled in the art may readily select a suitable precipitating type component and a suitable diluent type component to produce the desired product from a given charge stock. As a specific example, a mixture of about 60 to 80 volume percent of methylethylketone with from about 40 to 20 volume percent of an aromatic hydrocarbon such as benzene or toluene may be employed for treating the feed stocks of the present invention.

Dewaxing operations are preferably conducted at a temperature within the range of about −10° to about +20° F. and, more preferably, at a temperature within the range of about −10° to about +5° F. Wax deoiling or wax fractionation operations are preferably conducted at a temperature within the range of about 0° to about 120° F. which is higher than the dewaxing temperature employed. Thus, when dewaxing operations are conducted at a temperature of about −10° to about +5° F., it is preferable to conduct deoiling operations at a temperature within the range of about +10° to +20° F., depending upon the desired quality of deoiled wax.

The invention will be further illustrated with respect to the accompanying drawings wherein the sole FIGURE is a schematic flow sheet showing the manner in which dewaxing and deoiling operations are conducted in accordance with the present invention.

With reference to FIG. 1, a waxy lubricating oil fraction from a suitable source (not shown) such as a distillate fraction, a raffinate fraction, etc. is charged by way of a line **10** containing a pump **11** to a chilling zone **12**, the feed stock being charged to the chilling zone **12** in admixture with a suitable dewaxing solvent introduced into the system by way of a solvent charge line **14** controlled by a valve **15** leading to the feed stock charge line **10**.

The solvent mixed with the feed stock **10** by way of the line **14** is preferably a "wet" solvent containing less than about 3 volume percent of water. The amount of solvent to be employed should preferably be such that a dewaxed oil having desired predetermined physical properties for a lubricating oil base stock including a desired pour point and viscosity index is obtained in maximized yield at a maximized filtration rate. Thus, by way of specific example, for a distillate feed stock boiling within the range of about 440° to 750° F. at a pressure of 10 mm. of mercury and having a specific gravity of about 29° API, a pour point of about 115° F., and a viscosity of about 140 (SSU/130° F.), about 2½ volumes of a mixture of about 65 volume percent of methylethylketone with about 35 volume percent of toluene may be employed to provide a dewaxed oil having a pour point of about 10° F. and a viscosity index of about 83.

Within the chilling zone **12** the mixture of feed stock and solvent may be chilled to a suitable dewaxing temperature, such as a temperature of about 0° F. whereby there is formed a slurry of wax crystals associated with oil and solvent in a mother liquor consisting of oil and solvent. The slurry, to readily flow, should contain not more than about 50 volume percent of crystals. The slurry is discharged from chilling zone **12** by way of a line **16** leading to a turbulent isothermal filtration zone **18**. A recycle fraction obtained in a manner to be described is charged to the slurry line **16** by way of a recycle line **20** controlled by a valve **21**. While the turbulent isothermal filtration zone may be of any desired construction, it is preferably a zone containing "line filter" of the type disclosed in copending Moore and Royder application Serial No. 643,096, filed February 28, 1957, now U.S. Patent No. 2,914,456.

In the turbulent isothermal filtration zone **18**, a suitable amount of the mother liquor (e.g., about 20 to 60 volume percent of the slurry) is separated to thereby provide a mother liquor filtrate fraction and a concentrated slurry fraction. It is desirable that the amount of filtrate withdrawn should be such that the solids content of the concentrated slurry fraction is not so excessive as to impair the flowability of the concentrated slurry. Accordingly, it is preferable that the amount of slurry withdrawn be such that the concentrated slurry fraction contains from about 30 to 60 volume percent of solids.

The filtrate fraction, which is a solution of dewaxed oil in solvent, is withdrawn from the filtration zone **18** by way of a line **22** leading to suitable recovery facilities (not shown) for separating the dewaxed oil from the dewaxing solvent.

The concentrated slurry fraction is discharged from the filtration zone **18** by way of a line **24** leading to a second filtration zone **26**. The line **24** may, if desired, pass through a heating zone **23** for a purpose to be described. Prior to introduction into the zone **26**, the concentrated slurry fraction is diluted with a "dry" dewaxing solvent at the slurry temperature, the dry solvent, which may contain up to about 1 percent of water, being introduced by way of a charge line **28** controlled by a valve **29** leading to the line **24**. Although the amount of dry solvent to be employed may be varied within comparatively wide limits, it is generally preferable to employ from about 50 to 150 volume percent of solvent, based on the total amount of wet solvent charged by way of the line **14**. Still more preferably, from about 80 to 120 volume percent of dry solvent will be employed. The thus-diluted slurry fraction is separated into a second concentrated slurry fraction in the zone **26** which is discharged by way of a line **30** and into a filtrate fraction which is withdrawn by way of the line **20** for recycle in the described manner. The amount of filtrate separated from the slurry in the zone **26** will again be such that the slurry is concentrated without impairing its ability to flow. Alternately, all or a part of the filtrate fraction from the zone **26** may be discharged from the system for a purpose to be described by way of a discharge line **25** controlled by a valve **27**. Filtrate discharged by the line **25** may be charged to a zone for the separation of oil and solvent (not shown) which may be the same or different from the separation zone for the filtrate **22**.

From the foregoing description, it will be observed that, in accordance with this embodiment of the present invention, two filtration zones are employed in series, wet solvent being added to the charge stock for the first filtration zone of the series and dry solvent being added to the feed stock for the second filtration zone.

When a dewaxing operation is conducted in this fashion, there is a substantial reduction in the amount of solvent required. Thus, for the charge stock of FIG. 1, about 75 volume percent of dewaxed oil may be recovered from waxy oil while employing a total solvent circulation for the dewaxing step of about 2.5 barrels of solvent per barrel of feed stock. This constitutes a reduction of solvent circulation rate of about 40 percent as compared with the amount of solvent required to obtain an equivalent quantity of dewaxed oil employing a one-stage rotary or "line filter" operation.

Returning again to the drawing, the concentrated slurry fraction **30** may be treated to recover deoiled wax therefrom. When this is to be done, the slurry **30** may be mixed with a recycle fraction **38**, to be described, and the mixture may be warmed to about 0° to 100° F. depending on desired wax quality (e.g., about 30° F.) in a warming zone **32**. The warmed slurry is discharged from the warming zone **32** by way of a line **34** leading to a deoiling line filtration zone **36**. As a consequence, the slurry may contain about 10 to 20 percent of crystallized wax in a mother liquor containing about 10 to 20 volume percent of oil. About 15 to 25 volume percent of the mother liquor may be removed in the filtration zone **36** and discharged therefrom by way of a discharge line **42** controlled by a valve **43**. The thus-removed mother liquor constitutes a solution of high pour point dewaxed oil in solvent which may be charged to suitable oil and solvent recovery zones (not shown) or used as recycle to the dewaxing operation. In this latter situation, the filtrate is recycled to the charge line **10** by way of a recycle line **44** controlled by a valve **46**. The concentrated slurry fraction from the zone **36** is discharged therefrom by way of a line **48** leading to a rotary filter **40** of any suitable construction. The fraction, after being diluted with an amount of solvent added by way of line **49** sufficient to provide an easily filterable slurry is charged to the rotary filtration zone **40**. Within the rotary filtration zone **40** the wax is separated from the mother liquor to form a wax filter cake which is washed with fresh solvent introduced by way of a charge line **50**. The washed, deoiled wax, together with entrained solvent, is discharged from the rotary filtration zone **40** by way of a line **52** leading to a recovery zone (not shown). The mother liquor filtrate is recycled to the line **30** for the warming zone **32** by way of the line **38** in the described manner.

By way of specific example of the manner in which a paraffinic lubricating oil may be dewaxed in accordance with the present invention, a mixture of about 10 volumes of a wet (3% water) dewaxing solvent (a mixture of 65 volume percent of methylethylketone with 35 volume percent of toluene) with about 10 volumes of a wax-containing lubricating oil feed stock (80 volume percent of oil and 20 volume percent of wax) may be chilled to a temperature of about 0° F. in the chilling zone **12** to precipitate the wax. The slurry may be mixed with 10 volumes of a recycle slurry **20** containing about 92.3 percent solvent and about 7.7 percent oil in the line **16** and the resultant mixture may be charged to the first line filter zone **18**. Within the zone **18** the mixture may be separated into about 5 volumes of a first concentrated slurry fraction containing about 34.6 volume percent of solvent, about 25.4 volume percent of oil and about 40 volume percent of wax crystals and a filtrate containing about 25 volumes of a first filtrate fraction containing about 70 volume percent of solvent and about 30 volume percent of oil. The first filtrate fraction may be charged to a suitable recovery zone (not shown) of any desired conventional construction wherein the oil and solvent may be separated to provide make-up solvent and a low pour point dewaxed oil product.

About 10 volumes of dry solvent charged by way of the charge line **28** are added to the first concentrated slurry fraction **24** and the resultant mixture is charged to the second line filter zone **26**. Within the second line filter zone **26**, the charge mixture is separated into 10 volumes of the recycle filtrate fraction in line **20** described above and 5 volumes of a second concentrated slurry fraction in line **30** containing about 40 volume percent of wax crystals in a mother liquor containing about 50 volume percent of solvent and about 10 volume percent of oil. The second concentrated slurry fraction in line **30** may go to a wax-solvent recovery system (not shown) or be further processed in the wax-deoiling unit to recover deoiled wax in the described manner.

Thus, the 5 volumes of concentrated slurry may be mixed with about 14.4 volumes of recycle fraction from the line 38 to provide 19.4 volumes of a mixture containing about 87 volume percent solvent, about 2.3 percent of oil and about 10.7 percent of wax. The mixture is warmed to above 0° F. and turbulently isothermally filtered in the zone **36** to provide a filtrate fraction **42** and 3.2 volumes of a concentrated slurry fraction containing about 49.5 volume percent solvent, about 1 percent oil and about 49.5 percent wax. The fraction in the line **48** is then mixed with about 14.4 volumes of solvent to provide a slurry for the rotary filter **40** having about a 10:1 ratio of liquid to wax crystals. On rotary filtration of this slurry, a wax filter cake is obtained which is washed with about 1.6 volumes of solvent, whereby about 3.2 volumes of a filter cake containing about 50 volume percent of wax, about 50 percent of solvent (total oil content below about 0.3 percent) and 14.4 volumes of the recycle filtrate fraction containing about 93.5 volume percent solvent and about 0.5 percent oil.

Numerous advantages are obtainable through the provision of the process of the present invention. Thus, as mentioned above, a maximized recovery of dewaxed oil may be obtained with a minimized solvent circulation rate. In addition, several grades of dewaxed oil may be produced simultaneously. Thus, for example, a low pour point oil (0° to 10° F.), a medium pour point oil (30° to 70° F.), and a high pour point oil (30° to 70° F.) may be simultaneously obtained by separately recovering the oil in the filtrate fractions **22**, **25**, and **42**, respectively. In this situation, the slurry charged to the zone **26** will be heated in a warming zone **23** provided in the charge line **24** for the purpose of warming the slurry to a temperature sufficient to provide a "medium" pour point oil having a desired pour point. Still further, intermediate grades of lubricating oil may be produced by selectively discarding all or a portion of either the filtrate fraction **25**, the filtrate fraction **42**, or both. In this situation, of course, the amount of normally recycled filtrate (as described above) which is withdrawn from the system is replaced with an equivalent amount of fresh solvent.

As another example, an unexpected or temporary change in feed stock composition, which would normally result in an off-specification dewaxed oil, may be compensated for by selective filtrate discard, as set forth above.

Having described my invention, what is claimed is:

1. In a continuous method for the low temperature dewaxing of a paraffinic lubricating oil fraction wherein a solvent is employed, the improvement which comprises the steps of mixing the feed stock with fresh solvent, chilling the mixture to a temperature sufficient to form an initial slurry of crystallized oil-containing wax in a mother liquor comprised of dewaxed oil and solvent, passing the resultant slurry through a first turbulent isothermal filtration zone and turbulently isothermally removing from about 20 to 60 volume percent of said mother liquor contained therein to provide a first filtrate fraction and a first concentrated slurry fraction, recovering dewaxed oil from said first filtrate fraction, adding to said first concentrated slurry fraction an amount of fresh solvent substantially equivalent to the amount of mother liquor recovered from said first filtrate fraction to thereby form a diluted first slurry, heating said diluted first slurry to a temperature greater than that of said first turbulent isothermal filtration zone, passing said heated first slurry through at least a second turbulent isothermal filtration zone and turbulently isothermally removing from about 20 to 60 volume percent of the mother liquor therefrom to provide a second filtrate fraction and a second concentrated slurry fraction and recovering from said second filtrate fraction a dewaxed oil having a pour point greater than the pour point of the dewaxed oil recovered from said first filtrate fraction.

2. In a continuous method for the low temperature dewaxing of a paraffinic lubricating oil fraction wherein a solvent is employed, the improvement which comprises the steps of mixing the feed stock with fresh solvent, chilling the mixture to a temperature sufficient to form an initial slurry of crystallized oil-containing wax in a mother liquor comprised of dewaxed oil and solvent, passing the resultant slurry through a first turbulent isothermal filtration zone and turbulently isothermally removing from about 20 to 60 volume percent of said mother liquor contained therein to provide a first filtrate fraction and a first concentrated slurry fraction, recovering dewaxed oil from said first filtrate fraction, adding to said first concentrated slurry fraction an amount of fresh solvent substantially equivalent to the amount of mother liquor recovered from said first filtrate fraction, to thereby form a diluted first slurry, heating said diluted first slurry to a temperature greater than that of said first turbulent isothermal filtration zone, passing said heated first slurry through at least a second turbulent isothermal filtration zone and turbulently isothermally removing from about 20 to 60 volume percent of the mother liquor therefrom to provide a second filtrate fraction and a second concentrated slurry fraction, recovering from said second filtrate fraction a dewaxed oil having a pour point greater than the pour point of the dewaxed oil recovered from said first filtrate fraction, mixing a fourth filtrate fraction, as hereinafter defined, with said second concentrated slurry fraction to provide a diluted second slurry fraction, warming said second diluted slurry fraction to an extent sufficient to dissolve a desired amount of the oil initially contained in said wax into said mother liquor, passing said warmed second slurry through a third turbulent isothermal filtration zone and turbulently isothermally removing from about 20 to 60 volume percent of the mother liquor therefrom to provide a third filtrate fraction and a third concentrated slurry fraction, recovering from said third filtrate fraction a dewaxed oil having a pour point greater than the pour point of the dewaxed oil recovered from said second filtrate fraction, diluting said third filtrate fraction with solvent, charging said diluted slurry fraction to a rotary filtration zone and there resolving it into a waxy filter cake fraction and said fourth filtrate fraction as aforesaid.

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