

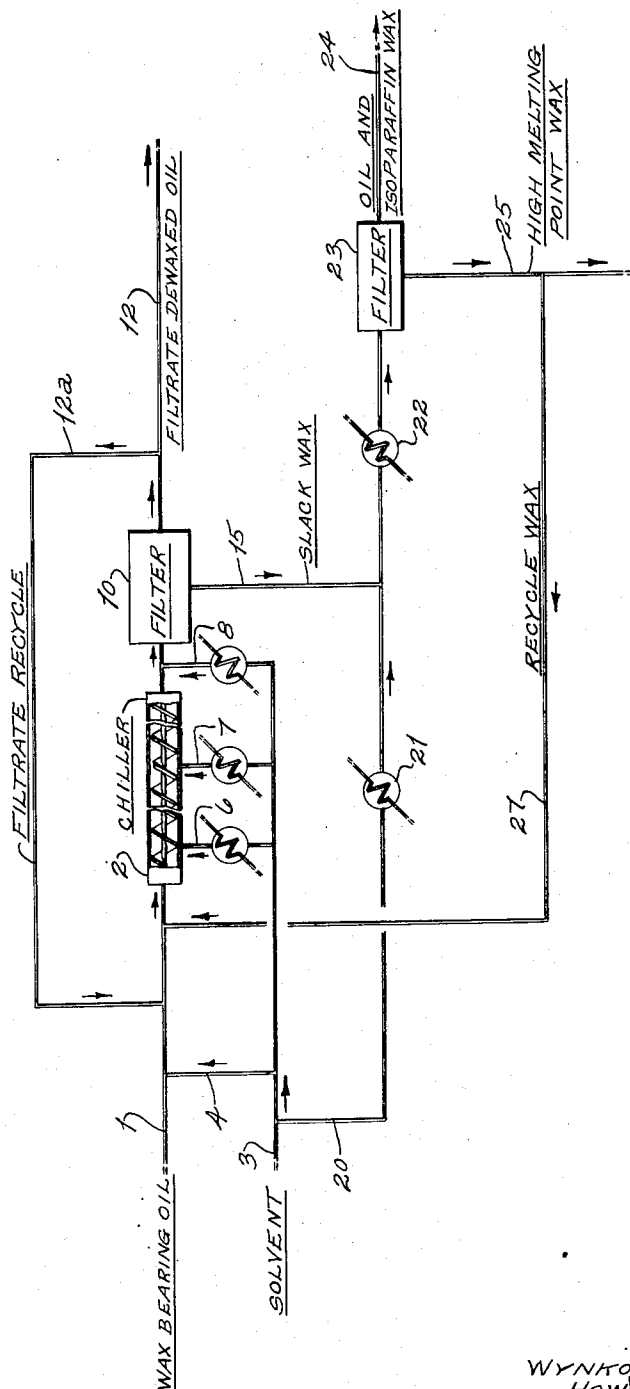
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SEPARATION OF OIL AND WAX BY CONTINUOUS FILTRATION

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SEPARATION OF OIL AND WAX BY
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This invention relates to the separation of oil and wax by continuous filtration wherein the oil-wax mixture is filtered in the presence of a solvent liquid at a temperature substantially below normal atmospheric.

The invention is concerned particularly with the dewaxing of wax-bearing lubricating oil stocks to produce a dewaxed oil of low pour test and to obtain a wax product highly normal paraffinic in character. In accordance with the invention the filtration is effected in the presence of a relatively small proportion of added wax which is normal paraffinic in character.

The invention is particularly advantageous from the standpoint of obtaining a product wax of low oil content and having a materially decreased content of soft wax or wax which is highly isoparaffinic in character.

The invention is to be distinguished from the dewaxing operation disclosed in U. S. Patent 2,248,498 granted to Howard H. Gross and Walter Smisloff. The patent in question discloses recycling to the filtering operation product wax and preferably a relatively low melting point portion of the product wax. The wax recirculation of this patent has for its object the increasing of the ratio of wax to oil in the type of wax-bearing oil feed from which the wax is normally deposited in the form of a cake which is too dense for effective filter operation.

In the practice of the present invention, the wax product obtained from the primary filter operation is further treated to separate therefrom a fraction of relatively high melting point and which is highly normal paraffinic in character. This high melting point fraction is recycled to the primary filtering operation. It is advantageous to employ a staged filtering operation as illustrated in the accompanying drawing.

This drawing comprises a diagram of flow adapted to the treatment of a wax-bearing lubricating oil distillate stock to obtain a dewaxed oil and also a high melting point wax rich in normal paraffinic constituents.

Referring to the drawing, a wax-bearing lubricating oil distillate derived from East Texas crude and having a Saybolt Universal viscosity of about 50-60 seconds at 210° F. is conducted from tankage through a pipe 1 to a chiller 2 which latter is a conventional tubular type.

The initial chilling of the wax-bearing oil flowing through pipe 1 may be effected in the substantial absence of solvent liquid if desired so as to facilitate the obtaining of the desired

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crystal structure, provision being made for addition of the solvent liquid in small increments at succeeding stages of temperature reduction. On the other hand, all of the solvent may be added prior to chilling if desired.

The solvent liquid may be a conventional dewaxing solvent such as a mixture comprising about 45% methylethyl ketone and 55% commercial benzol and is conducted from a source not shown through a pipe 3. In the event that it is desired a portion of the solvent liquid may be diverted through branch pipe 4 for commingling with the incoming stream of wax-bearing oil prior to chilling in the proportion of about 0.1 to 0.5 volume of solvent liquid per volume of oil. This initial dilution may be effected by addition of recycled filtrate as will be referred to later.

The wax-bearing oil stream partially diluted with solvent liquid, or in the alternative, in the substantial absence of solvent, flows into the chiller 2. After passage through a portion of the chilling system cold solvent liquid is diverted through pipe 6. If desired, an additional quantity of the solvent liquid may be added at a succeeding point in the chiller, as for example, through pipe 7.

The wax-bearing oil and solvent liquid leaves the chiller at a temperature of about -15° F. At this point, it is mixed with the remainder of the solvent separately chilled to approximately this temperature, this further quantity of solvent being added through branch pipe 8.

The wax-bearing mixture diluted with about 3-4 volumes of solvent liquid per volume of wax-bearing feed oil is then passed into a continuous rotary drum vacuum filter 10 of conventional design.

The filter is operated at about -15° F. and provision is made, although not shown, for washing the filter cake in situ with additional solvent liquid.

The filtrate obtained comprises dewaxed oil dissolved in the main body of solvent liquid. This filtrate is discharged through pipe 12 to a solvent recovery unit not shown wherein the solvent is stripped from the oil leaving a dewaxed oil having a pour test of about minus 10 to minus 15° F.

The filter cake is continuously discharged from the filter through pipe 15 and comprises slack wax mixed with solvent liquid. The solvent liquid may amount to 50 to 80% by volume of the filter cake. The wax, amounting to about 40% by volume of the lubricating oil distillate,

will comprise a mixture of isoparaffinic and normal paraffinic constituents.

This slack wax is mixed with a quantity of solvent liquid and drawn through branch pipe 20 and heat exchanger 21. The mixture of solvent and slack wax passes through heat exchanger 22 and is brought to a temperature of about 30° F. Sufficient solvent is added to obtain a mixture comprising about 7 volumes of solvent per 1 volume of slack wax.

This mixture is then passed through a filter 23 similar to filter 10. The filtrate is discharged through pipe 24 and will comprise low melting point wax rich in isoparaffin constituents dissolved in the main body of solvent liquid. It is conducted to solvent recovery means not shown.

The filter cake is discharged through pipe 25 and comprises high melting point wax having a normal paraffinic character. It is also mixed with solvent liquid in the proportion of about 1 to 4 volumes of solvent per volume of wax. A portion thereof is diverted through pipe 27 while the remainder is discharged and the solvent recovered therefrom. This wax will have a melting point of about 145° F. and will amount to about 60% by volume of the slack wax charged to the filter 23. It will consist substantially entirely of normal paraffins. The recycled wax passing through pipe 27 is injected into the wax-bearing oil feed stream flowing through the pipe 1. The amount so recycled ranges from about 5-20% by volume of the feed oil charge.

As previously mentioned, a portion of the primary filtrate may be recycled through the primary filtering operation. Thus a portion of the filtrate may be diverted from pipe 12 through branch pipe 12a and injected into the wax-bearing oil feed stream as indicated. It may be injected into this stream prior to introduction to the chiller 2 or after partial chilling or, if desired, after the feed stream leaves the chiller and prior to introduction to the filter.

The amount of filtrate recycle will depend upon the character of the feed oil undergoing treatment and also to a small extent upon the amount of wax recycle. In any case, it is desired to adjust the amount of filtrate recycle so that the relative proportions of solid phase and liquid phase in the chilled mixture undergoing filtration in the filter bowl will be about 1 volume of solid phase to from about 12 to 25 volumes of liquid phase.

It will be understood that the temperature prevailing in the filter 10 may be varied depending upon the degree of dewaxing desired but will usually be in the range of about 0 to -25 or -30° F. Likewise, the temperature prevailing in the filter 23 will vary depending upon the melting point of the wax product desired and will usually range from about 20 to 40° F.

Other conventional dewaxing solvent liquids may be employed. It is usually preferred to employ a mixture of a low molecular weight aliphatic ketone and an aromatic hydrocarbon such as benzene, toluene, etc. Other wax antisolvent liquids than these ketones may be employed, however, and likewise other oil solvent liquids besides aromatic hydrocarbons may be used. Single solvent liquids may be used, as for example, petroleum naphtha or liquefied normally gaseous hydrocarbons such as propane or butane or mixtures thereof.

While mention has been made in the detailed description of charging a lubricating oil stock derived from East Texas crude, it will be understood

that the invention has application to the treatment of other stocks such as derived from Pennsylvania crudes.

In any case, it is contemplated effecting the primary filtering operation in the presence of an added wax which consists essentially of normal paraffins and which is substantially free from isoparaffins. By recirculating such wax the oil content of the product wax may be reduced by as much as 25 to 50% of that which is present in the product when the wax recycling of this invention is not employed. Moreover, the product will be richer in normal paraffins.

Normal paraffin wax hydrocarbons regardless of molecular weight exhibit the same refractive index value at their melting point namely 1.4365. Isoparaffinic wax hydrocarbons have a much higher refractive index value. Thus the slack wax removed from the filter 10 through pipe 15 when no wax is being recycled may have a refractive index of about 1.4415 to 1.4445. On the other hand, when wax is being recycled through pipe 27 the wax product removed from filter 10 will have a refractive index of about 1.4385 to 1.4400.

Obviously many modifications and variations of the invention as above set forth may be made without departing from the spirit and scope thereof, and therefore only such limitations should be imposed as are indicated in the appended claims.

We claim:

1. In the separation of oil and wax from mixtures thereof, said wax comprising both normal and isoparaffinic constituents, the method comprising continuously passing a stream of said oil-wax mixture with solvent liquid, at reduced temperature in the range of about 0° F. and below to a primary zone of filtration, effecting said filtration of said mixture of reduced temperature thereby forming a filtrate of dewaxed oil dissolved in solvent liquid and a wax cake comprising said wax, discharging said filtrate, removing said wax cake, subjecting said removed wax to filtration in the presence of solvent liquid at a temperature substantially above said reduced temperature and below normal room temperature in a secondary filtration, forming a secondary filtrate containing soft isoparaffinic wax and a wax cake consisting essentially of normal paraffin wax constituents, and recycling a portion of said normal paraffinic wax to the wax mixture passing to said primary filtration.

2. The method according to claim 1 in which the feed oil-wax mixture is a wax-bearing distillate lubricating oil stock.

3. The method according to claim 1 in which the feed oil-wax mixture is a wax-bearing distillate lubricating oil stock and the recycled wax amounts to about 5 to 20% by volume basis said distillate.

4. In the dewaxing of wax-bearing lubricating oil stock containing both normal and isoparaffin wax constituents to produce dewaxed oil of low pour point and to obtain a wax of highly normal paraffinic character by a process involving chilling and continuous filtration of a chilled mixture of said wax-bearing oil and dewaxing solvent liquid to form a filtrate containing oil and a filter cake of wax, the wax content of said filter cake as normally obtained being characterized by having a refractive index in the range about 1.4415 and higher, the improvement which comprises effecting crystallization of both normal and isoparaffin wax constituents in the presence of a small proportion of added wax con-

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sisting essentially of normal paraffins and having a refractive index not in excess of about 1.4365, and effecting the filtration of the resulting mixture containing added wax at a temperature in the range about 0° F. and below, thereby obtaining a filter cake the content of which has a refractive index in the range below about 1.4400.

5. In the dewaxing of wax-bearing lubricating oil stock containing both normal and isoparaffin wax constituents to produce dewaxed oil of low pour point and to obtain a wax of highly normal paraffinic character by chilling and continuous filtration of the resulting chilled mixture, the process which comprises effecting said filtration at a temperature in the range 0° F. and below thereby forming a filtrate of dewaxed oil and a filter cake of normal and isoparaffin wax constituents, forming a fluid mixture of said filter cake with solvent liquid, filtering said last mentioned mixture at a temperature substantially above 0° F. and such that a filtrate-containing isoparaffin wax constituent is obtained and a filter cake consisting essentially of normal paraffin wax constituents is obtained, and recycling a small proportion of normal paraffin wax constituents derived from said last mentioned filter cake to the initial feed mixture prior to the first mentioned filtration.

6. In the dewaxing of wax-bearing lubricating oil stock containing both normal and isoparaffin wax constituents to produce dewaxed oil of low pour point and to obtain a wax of highly normal paraffinic character by a process involving chilling and continuous filtration of a chilled

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mixture of said wax-bearing oil and dewaxing solvent liquid to form a filtrate containing dewaxed oil and a filter cake of wax, the wax content of said filter cake as normally obtained being characterized by having a refractive index in the range about 1.4415 and higher, the improvement which comprises effecting crystallization of substantially all of the normal and isoparaffin wax constituents in the presence of a small proportion of added wax consisting essentially of normal paraffins and having a refractive index not in excess of about 1.4365, and effecting the filtration of the resulting mixture containing added wax at a temperature in the range about 0° F. and below in a single filtering step and thereby obtaining a filter cake the wax content of which has a refractive index in the range below about 1.4400.

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