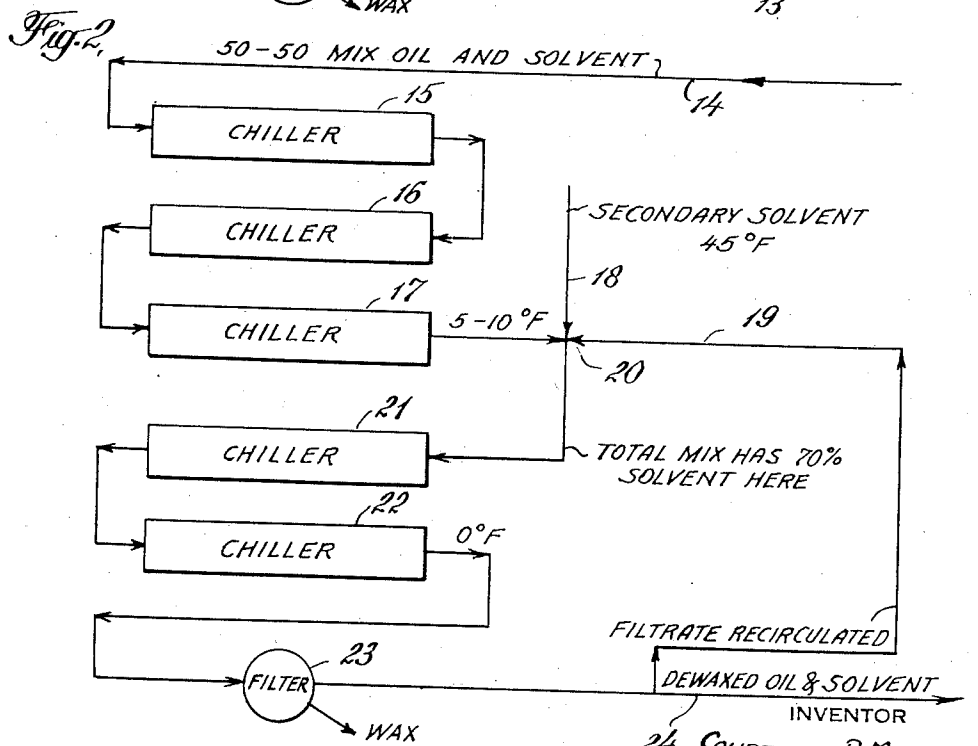
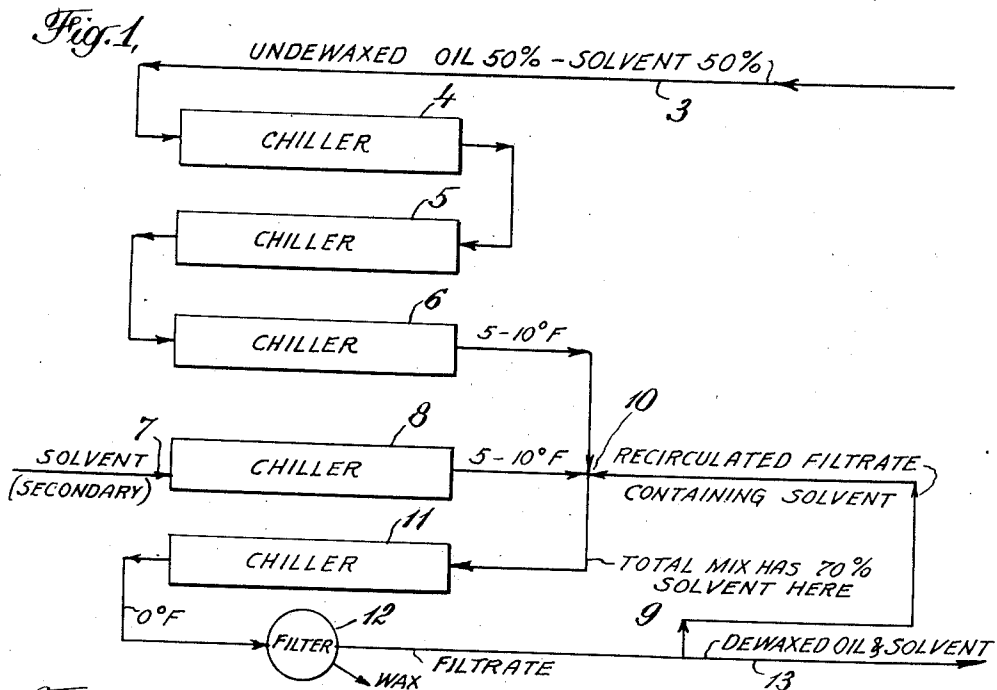


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C. D. MARSHALL
METHOD OF DEWAXING OIL

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METHOD OF DEWAXING OIL

Courtenay D. Marshall, Beaumont, Tex., assignor
to Socony-Vacuum Oil Company, Incorporated,
New York, N. Y., a corporation of New York

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3 Claims. (Cl. 196--18)

This invention has to do with the dewaxing of petroleum lubricating oil fractions by methods currently described as "solvent dewaxing."

Petroleum fractions of the nature of distillates and residues and mixtures thereof, containing waxy materials, are normally treated to remove the wax therefrom by adding to the waxy oil a diluent material, chilling the mixture to a temperature at which the wax, or a substantial portion thereof is precipitated, and filtering or otherwise physically separating the wax from the oil. Those methods in which the diluent used is one tending to exert a preferential solvent action for oil over waxy material are commonly known as "solvent dewaxing" methods. One very widely used version of this method makes use of a "solvent-anti-solvent" mixture of an oil solvent such as benzol and/or toluol and a wax anti-solvent such as a ketone, for example acetone or methyl-ethyl ketone. The usual proportions of this "B-K" solvent are 40-60% ketone, the rest being benzol and/or toluol. While this invention is described hereinafter with respect to a solvent of this character, the principle disclosed herein is widely applicable and the limitation in description is merely by way of example.

Prior methods of practice have developed two principles of operation. One is that of the use of a secondary solvent. That is, only a portion of the solvent present in the filtration stage is initially added to the waxy oil. This mixture is then chilled to precipitate a substantial portion of the wax, after which a further portion of pre-chilled solvent is added to the mix, the whole being then chilled to separation temperature level and the wax separated. The second principle is that of filtrate recycle, wherein a portion of the filtrate, comprising solvent and dewaxed oil from the wax separation operation is added to the incoming waxy oil and solvent mixture, usually at a point intermediate in the chilling, to again pass through the final chilling and wax separation steps.

Both of these operations have advantages from increased ability to control the physical nature of the filter cake, increased yield of dewaxed oil from a given stock, and the like, and both are widely used, either together or singly.

This invention has as its object the provision of a modified dewaxing procedure of this kind, using the secondary solvent addition, with or without filtrate recycle, with certain modifications hereinafter explained, whereby entirely unexpected increases in the yield of dewaxed oils from a given stock may be obtained.

In the previously practiced process, the waxy oil charge was mixed with an approximately equal volume of benzol-ketone solvent and the mixture chilled to about 5-10° F. A separately chilled portion of solvent, free from oil, pre-chilled to about 5-10° F. was then added, and at the same time, recirculated filtrate comprising about 72% of solvent, the remainder being wax-free oil, the recirculated filtrate being at a temperature of about 5° F. was added, the proportioning being such that the amount of solvent in the total resulting mix was about 70%. The whole mix was then chilled to the final dewaxing temperature of 0° F. and the precipitated wax was separated.

According to the modified process, the same starting mixture as before is chilled as before added, and also, the same amount of recirculated filtrate, at the same temperature, is then added, and also, the same amount of secondary solvent is added at this point, with this distinction. The secondary solvent is not pre-chilled, but is added at a temperature of about 45° F. The total mixture is then chilled to 0° F. and precipitated wax is separated. When this method was adopted, the surprising discovery was made that this comparatively small change, using the same stock, same filtering temperature, same solvent in the same proportions, and same equipment, enabled the operation of the filter at approximately a 20 percent higher throughput rate, while increasing the yield of dewaxed oil from the operation by about 5%.

These increases are significant in amount, since not only is the amount of dewaxed oil which can be recovered from a given stock increased, but also a major increase in the dewaxing capacity of a given equipment is gained, the two together amount to something like a twenty-five percent increase in the dewaxed oil production of the plant to which it is applied.

While I do not attempt to base this invention upon any conjectural theory of operation, it would appear that the change in temperature of the secondary solvent at the point of its application effects a major change in the character of the precipitated wax masses. Noting that in both processes a substantial portion of the wax is already precipitated at the time of the addition of secondary solvent, and that in the older process the secondary solvent is pre-chilled to the same temperature of the mix to which it is added, and would not presumably have any effect other than the precipitation of more wax, due to its added preferential solvent effect, it

appears possible that in the new process, the added relatively warm solvent may serve first to dissolve some of the already precipitated wax which would result, upon further chilling, in a more open structure of wax crystals capable of giving the improved results. Whatever the explanation may be, the results are greatly improved, as may be noted by consideration of the following comparative operations.

Old operation.—A waxy stock consisting of a furfural refined distillate from East Texas crude having a viscosity 95 S. U. V. at 130° F. was blended with an equal proportion, by volume of solvent mixture consisting of 45% commercial benzol and 15% of commercial toluol and 40% of methyl ethyl ketone. The mixture was chilled to 7° F. and there was added to it 120% by volume based on waxy stock of secondary solvent of the same composition as the first solvent, which secondary solvent was pre-chilled to a temperature of 10° F. At the same time there was added, to the extent of 110% based upon the waxy stock, recycled filtrate, containing 72% of solvent and 28% of dewaxed oil. The total mixture, now containing 70% of solvent and having a temperature of 7° F., was then chilled to 0° F. and filtered. The filter rate was 2.18 gallons of dewaxed oil per square foot of filter surface per hour and the operation succeeded in recovering 76.0% of dewaxed oil based upon waxy charge stock.

New operation.—The same waxy stock, namely, a furfural refined distillate from East Texas crude having a 95 S. U. V. at 130° F. was blended in equal amounts by volume with the same solvent, and chilled, as before to 7° F. Then there was added to it recycle filtrate, to the extent of 110% based upon waxy stock, the filtrate containing 72% of solvent, the remainder being dewaxed oil, the filtrate having a temperature of 5° F., all these conditions being substantially the same as before. At the same time, there was added secondary solvent, in substantially the same amount as before, excepting that this secondary solvent was not pre-chilled, being added at a temperature of 45° F. The total mixture, containing 70% of solvent and having a temperature of 17° F. was then chilled to 0° F. and filtered. The filter rate was 2.63 gallons of dewaxed oil per square foot of filter surface per hour and the operation succeeded in recovering 80.3% of dewaxed oil based upon waxy charge stock.

To clearly contrast this new operation with the old, a drawing is provided herewith in which Figure 1 shows the old operation and Figure 2 the new, both in diagram form. In Figure 1, waxy stock blended with solvent enters at 3, passes through chillers 4, 5, 6, and is chilled to 5–10° F. Secondary solvent enters at 7 and is chilled in chiller 8 to the same temperature of 5–10° F. Chilled blend, chilled secondary solvent and recirculated filtrate from 9 are mixed at 10 and proceed through chiller 11 to be chilled to 0° F. Wax is separated in filter 12, and filtrate is removed from process through 13, a portion being recirculated through 9.

In Figure 2, waxy stock blended with solvent enters at 14, passes through chillers 15, 16, 17, and is chilled to 5–10° F. Secondary solvent, not pre-chilled, from 18 and recirculated filtrate from 19 are mixed with the chilled blend at 20. The mixture proceeds through chillers 21 and 22 to be chilled to 0° F. and is filtered at 23 to

separate precipitated wax. Filtrate is withdrawn at 24, a portion being recirculated at 19.

Certain variations are possible without departing from the spirit of the invention. Other solvents and diluents than that shown may be utilized. The process of separation need not be that of filtration which is shown, but may be any process mechanically appropriate for the separation of the precipitated wax. The specific proportions noted, while appropriate for the particular stock of the example will vary from stock to stock and from solvent to solvent. The amount of the total solvent added must be sufficient to preserve in the mixture a proper fluidity, at dewaxing temperature, to permit wax removal. In general, with B-K types of solvents and the usual dewaxing stocks, the amount of solvent in the final mix flowing from chillers to wax separation will be from about 65% to about 93% of the mix. Similarly, the amount of solvent originally blended with the incoming waxy stock, before chilling, will be from about 65% to about 550% by volume of the waxy stock. The primary solvent should be present in amount sufficient to maintain the mixture in fluid condition at those temperatures, near but above the final dewaxing temperature, which are attained in the first chilling. The remainder of the solvent present in the final mix will be made up from secondary solvent and recirculated filtrate. In some circumstances no recirculated filtrate will be used. In general, the proportions will be from about 80% to about 450% of secondary solvent, and from about 50% to about 750% of recirculated filtrate, both by volume based upon waxy stock originally charged.

All temperatures in the system will vary of course with solvent used, stock being dewaxed, and extent of dewaxing. Those shown are typical of present commercial practice in dewaxing with B-K solvents. The temperature at which the secondary solvent is added will be substantially above that of the chilled blend of oil and primary solvent to which it is added. This will also vary with stock, solvent, and proportions, but in general this temperature will be from about 20° F. to about 60° F. above the chilled blend temperature. In the present instance it is about 40° above the chilled blend temperature.

The constitution of the recirculated filtrate is of course dictated by the final proportions of the mix going from the chillers to the wax separation step, modified by the removal of wax, and in some instances by the inclusion of variable amounts of wash solvents with the filtrate. In general it will contain from about 67% to about 95% of solvent, the remainder being dewaxed oil.

This recirculated filtrate will be added usually at a temperature within about 3° F. of (generally below) that of the blend, this temperature ranging from about –15° F. to about 15° F.

Due to the variable addition of secondary solvent and of recirculation filtrate and the variable temperatures permissible in such additions, it is possibly better to rephrase the description of the process in the following terms: Starting with an initial blend of from about 40% to about 85% of solvent and from about 60% to about 15% of oil, that blend is chilled to a temperature of from about –12° F. to about +18° F., sufficient to precipitate a substantial amount of the wax present. Then sufficient solvent is added from the sources indicated to bring the percent by volume of solvent in the solvent-oil-wax mix to from about 65% to about 93%, the additions being

at such temperatures as to bring the total mix to a temperature of from -5° F. to about 35° F. Then the total mix is again chilled to the final wax precipitation temperature and the precipitated wax is separated.

I claim:

1. That method of dewaxing a waxy petroleum oil stock comprising the steps of admixing with the waxy oil a dewaxing solvent in amount sufficient to maintain the mixture in fluid condition at temperatures near but somewhat above final dewaxing temperature, chilling the mixture to a temperature near but above the final dewaxing temperature, adding a second portion of solvent which is at a temperature substantially above that of the chilled mixture, chilling the so-formed mixture to final dewaxing temperature and separating the wax therefrom.

2. That method of dewaxing a waxy petroleum oil stock comprising the steps of admixing with the waxy oil a dewaxing solvent in amount sufficient to maintain the mixture in fluid condition at temperatures near but somewhat above final dewaxing temperature, chilling the mixture to

a temperature near but above the final dewaxing temperature, adding a second portion of solvent which is at a temperature from about 20° F. to about 60° F. above that of the chilled mixture, chilling the so-formed mixture to final dewaxing temperature and separating the wax therefrom.

3. That method of dewaxing a waxy petroleum oil stock comprising the steps of admixing with the waxy oil a dewaxing solvent in amount sufficient to maintain the mixture in fluid condition at temperatures near but somewhat above final dewaxing temperature, chilling the mixture to a temperature near but above the final dewaxing temperature, adding a second portion of solvent in amount sufficient to render the resulting mixture fluid at the final dewaxing temperature, and a temperature sufficient to raise the resulting mix to from about 5° F. to about 20° F. above the temperature resulting from the first chilling, then chilling the resulting mix to the final dewaxing temperature and separating the precipitated wax.

COURTENAY D. MARSHALL.

CERTIFICATE OF CORRECTION.

Patent No. 2,284,607.

May 26, 1942.

COURTENAY D. MARSHALL.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 1, second column, line 18, for "added, and also" read --to about 5-10° F.--; page 3, second column, line 18, claim 3, after "and" insert --at--; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 22nd day of September, A. D. 1942.

(Seal)

Henry Van Arsdale,
Acting Commissioner of Patents.