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TWO-STAGE SOLVENT DEWAXING OF LUBRICATING OIL

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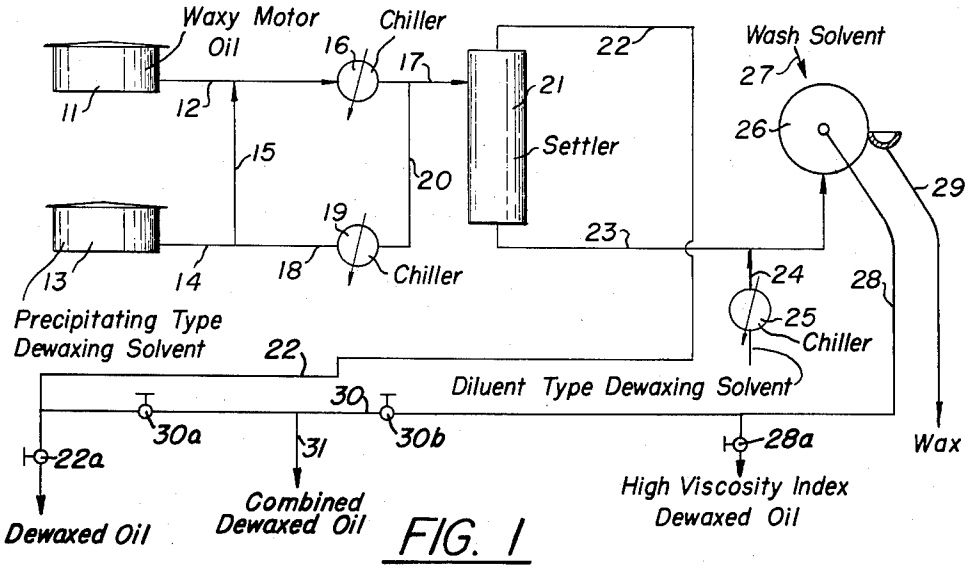


FIG. 1

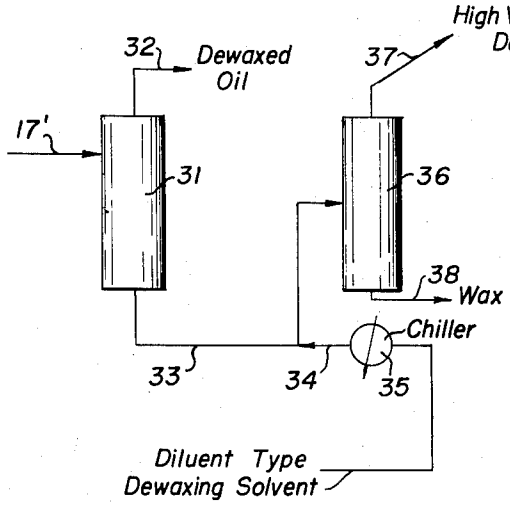


FIG. 2

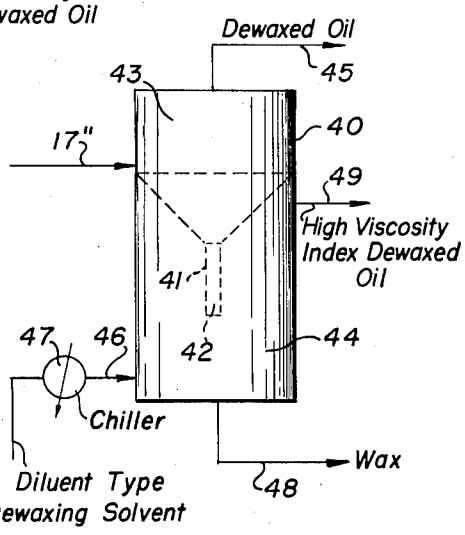


FIG. 3

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TWO-STAGE SOLVENT DEWAXING OF LUBRICATING OIL

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5 Claims. (Cl. 196—18)

The present invention is directed to a method for solvent dewaxing lubricating oil.

It is well known to the art to remove wax from a waxy oil by dissolving the waxy oil in solvent, chilling the resultant solution to cause wax to precipitate and then separating the precipitated wax by some suitable means such as filtration or gravity settling. The present application is directed to an improved method for solvent dewaxing oil.

The practice of the present invention will now be described in detail in conjunction with the accompanying drawing in which:

Fig. 1 is in the form of a diagrammatic flow sheet illustrating one procedure for practicing the present invention;

Fig. 2 is in the form of a diagrammatic flow sheet illustrating another procedure for practicing the present invention; and,

Fig. 3 is in the form of a diagrammatic flow sheet illustrating another procedure for practicing the present invention.

In the dewaxing procedure of the present application two separating steps are employed for separating the oil from the wax. The procedure involves the use of two dewaxing solvent components, one of the components being a precipitating type component and the other being a diluent type component. For the first step the solvent mixture composition is adjusted so that the miscibility temperature is substantially above the selected dewaxing temperature and in a first separating step at the dewaxing temperature a solvent rich phase is separated from a mixture of a solvent lean phase and a wax solid phase. To the mixture of solvent lean phase and wax solid phase is added sufficient diluent type solvent component to dissolve and wash substantially all of the oil from the wax crystals and the resultant solution is then separated in a second separating step into a wax phase and a dewaxed oil phase. The dewaxed oil fractions obtained from the two separating steps may be combined or may be used as separate fractions.

In carrying out the procedure described in the preceding paragraph in the first step it is desirable that the miscibility temperature be at least 40° F. higher than the selected dewaxing temperature thereby insuring that at the dewaxing temperature a clear layer consisting of from 25 to 50% of the total will form in about five minutes upon settling under quiescent conditions. It will also be found preferable to conduct the first separating step by gravity settling.

Specific examples of solvents and solvent mixtures which may be used in the practice of the present invention are as follows: The precipitating type component may be a ketone and the diluent type component may be an aromatic material. Specific ketones are acetone, methyl ethyl ketone, methyl normal propyl ketone and diethyl ketone or mixtures thereof while the aromatic materials serving as the diluent type component may be benzol or toluol or mixtures thereof. As another

group of solvent mixtures the precipitating type component may be ethylene dichloride used in conjunction with a diluent type component which may be benzene or chloroform or carbon tetra-chloride or mixtures thereof. As another example, the precipitating type component may be furfural and used in conjunction with benzol as the diluent type component. It is to be understood in the specific examples listed that the precipitating type component and the diluent type component are not necessarily pure. As a specific example, the precipitating type component may consist of a major amount of methyl ethyl ketone and a minor amount of benzol while the diluent type component may consist of a minor amount of methyl ethyl ketone and a major amount of benzol. Similarly, when using other pairs of components, mixtures may be employed for each component. It will be apparent to a workman skilled in the art that many other well known dewaxing solvents and solvent mixtures have the requisite characteristics for use as precipitating type components and diluent type components. A workman skilled in the art may readily select a suitable precipitating type solvent or solvent mixture for use with a suitable diluent type solvent or solvent mixtures in the practice of the present invention.

The invention will now be further described with reference to the drawing.

Turning now specifically to Fig. 1 a waxy motor oil is withdrawn from storage vessel **11** by line **12**. A precipitating type dewaxing solvent (which may consist of a mixture of compounds) is withdrawn from storage tank **13** through line **14** and a portion is passed through branch line **15** where it is used to dissolve waxy motor oil in line **12**. The solution of waxy motor oil in solvent passes to a chilling step **16** where it is chilled to a suitable dewaxing temperature (which is substantially below the miscibility temperature) and is withdrawn through line **17** where additional precipitating type solvent which is passed by line **14** through branch line **18** and chilled to dewaxing temperature in chiller **19** is added thereto by line **20**. The resultant solution discharges into first settling zone **21** which is shown as a form of a settler. The chilled waxy oil solution discharged into settling zone **21** consists of three phases, a solvent rich liquid phase, a solvent lean liquid phase and a wax solid phase. The settling zone **21** is maintained under quiescent settling conditions whereupon from 25% to 50% of a clear layer consisting of the solvent rich phase forms in a short time. A mixture of solvent lean phase and wax solid phase forms the other layer. The solvent rich phase layer is removed from vessel **21** through outlet line **22** and may be withdrawn as a desired dewaxed oil fraction. The mixture of solvent lean phase and wax solid phase is withdrawn through line **23** and has added thereto an additional amount of chilled diluent type solvent introduced through line **24** containing chiller **25**. The resultant solution is then discharged to a second separating zone shown as a rotary type filter **26**. The use of such filters is well known. If desired the wax cake on the filter may be washed with solvent introduced through line **27**. A dewaxed oil fraction is withdrawn from the filter through outlet line **28** and a wax fraction is withdrawn through outlet line **29**. The dewaxed oil phase in line **22** and the dewaxed oil phase in line **28** may be combined in line **30** controlled by valves **30***a* and **30***b* and withdrawn by line **31** on closing valves **22***a* and **28***a*.

Another procedure for carrying out the present invention is illustrated in Fig. 2. In Fig. 2 the mixing of the solvent with the dewaxed oil and preliminary chilling involving steps **11** to **20** are identical with that of Fig. 1 and are indicated by like reference numerals. However, the chilled mixture consisting of solvent rich phase, solvent lean phase and wax solid phase from line **17'** (cor-

responding to line 17 of Fig. 1) is discharged into first separating zone 31 where it is maintained under quiescent settling conditions to form a clear upper layer consisting of solvent rich phase and a lower layer consisting of a mixture of solvent lean phase and wax solid phase. The solvent rich phase is removed through outlet line 32 as dewaxed oil. The mixture of solvent lean phase and wax solid phase is withdrawn through outlet line 33 where it has added thereto an additional amount of diluent type solvent (which may consist of a mixture of compounds) introduced through inlet line 34 containing chiller 35. A sufficient amount of diluent type solvent is added to the mixture of solvent lean phase and wax solid phase to dissolve and wash substantially all of the oil from the wax crystals. The resultant solution is discharged into a second settling zone 36 in which the mixture is maintained under quiescent conditions to allow the wax crystals to settle from the solution of wax free oil. A solution of the wax free oil is withdrawn from settler 36 through outlet line 37 and the wax fraction is withdrawn through outlet 38.

Another mode for practicing the present invention is illustrated in Fig. 3. In Fig. 3 the preliminary steps of addition of solvent to waxy oil and chilling carried out in steps 11 to 20 are identical to the like steps of Figs. 1 and 2 and are designated by identical reference numerals. An elongated vessel 40 is divided by section 41 having a central passage 42 so that the vessel forms an upper settling zone 43 and a lower settling zone 44 with the two zones being connected by passage 42. The mixture of solvent rich oil phase, solvent lean oil phase and wax solid phase is discharged from line 17" (corresponding to line 17 of Fig. 1) into upper settling zone 43 which is maintained under quiescent settling conditions. The settling zone 43 serves as a first separating zone from which solvent rich phase is withdrawn through outlet line 45 as a dewaxed oil fraction. A mixture of solvent lean oil phase and wax crystals settles to the lower portion of settling zone 43 and passes downwardly through outlet 42 into lower settling zone 44. Diluent type solvent (which may consist of a mixture of compounds) is introduced into the lower settling zone 44 by means of inlet line 46 and is chilled to dewaxing temperature by chiller 47 before it enters said zone 44. The diluent type solvent is added into settling zone 44 in such an amount as to dissolve and wash substantially all of the oil from the wax crystals in said zone. The substantially oil free wax is withdrawn from the zone through outlet line 48 and a high viscosity index oil solution free from wax is withdrawn through outlet line 49.

An example of suitable operating conditions will now be given with reference to the drawing. The waxy motor oil in storage tank 11 may consist of 78% oil and 22% wax at 150° F. while the precipitant type solvent from storage tank 13 may consist of 85% methyl ethyl ketone and 15% toluol at 150° F. 100 parts of the precipitant type solvent mixture is passed through line 15 to dissolve 100 parts of waxy motor oil. The miscibility temperature is approximately 50° F. and the selected dewaxing temperature is 0° F. The solution is then chilled to the selected dewaxing temperature of 0° F. and has added thereto 200 parts of additional precipitant type solvent cooled to 0° F. the resultant slurry discharging into settler 21. From settler 21 a solution of dewaxed oil is removed through line 22 which consists of 12.7 parts oil and 130 parts solvent. From the bottom of the settler a mixture is removed which consists of 22 parts wax, 65.3 parts oil and 170 parts solvent at 0° F. To this mixture is added 44 parts of 100% toluol at 0° F. through line 24 to produce a solvent mixture having a composition of 68% methyl ethyl ketone and 32% toluol which substantially completely dissolves the oil in the solvent lean phase and washes all of the oil from the wax crystals. The resultant mixture is then filtered in rotary filter 26.

The advantages of the present invention are further

shown by three different runs with the results tabulated hereafter in the table. Run 1 was carried out as a blank to represent the prior art, and runs 2 and 3 were carried out in accordance with the present invention. That is, run 1 was carried out in a single stage while runs 2 and 3 were carried out in two stages. It will be noted that the precipittaing type solvent employed for initially dissolving the waxy oil consisted of a solution of methyl ethyl ketone, toluol and water, while toluol was used for the diluent type solvent subsequently added.

In the table it will be noted that while the filtering rate based on the wax separated was 24 lbs./sq. ft./hr. in run 1, the filter rate was increased to 49 lbs./sq. ft./hr. and 46 lbs./sq. ft./hr., respectively, in runs 2 and 3 which were carried out in accordance with the present invention.

Table

Run	1	2	3
Solvent Composition:			
Percent Methyl ethyl ketone	68	85	90
Percent Toluol	32	15	10
Percent Water added	0.6	1.65	2.1
Miscibility temperature, ° F	−10	+48	+70
Primary dilution, volumes @ 150° F	1.0	1.0	1.0
Secondary dilution, volumes @ 0° F	1.5	2.0	2.0
Percent clear layer in 5 minute settling		33	36
Yield of oil from settling		12.7	16.7
Percent oil in clear layer		9.8	7.7
Tests—viscosity SSU @ 100° F		680	834
viscosity SSU @ 210° F		64.1	67.0
Viscosity Index		59	44
Pour, ° F		0	−5
Filtering cold settled wax—adjusted solvent composition to 68% methyl ethyl ketone by adding toluol:			
Filtering Dilution	2.5	3.2	2.5
Oil Rate, gals./sq. ft./hr	5.6	11.8	6.6
Wax Rate, lbs./sq. ft./hr	24	49	46
Yield of oil in filtering (on original charge)	63	55.3	42
Yield of wax in filtering (on original charge)	37	32	41.3
Oil Tests:			
Viscosity @ 100° F.—SSU	490	464	435
Viscosity @ 210° F.—SSU	60.2	59.2	58.7
Viscosity Index	83	85	90
Pour	0	10	10
Wax test: percent oil	37	32	44

While I have disclosed specific examples illustrating the practice of the present invention, it will be obvious to a workman skilled in the art that various changes may be made in the solvent compositions, temperatures of operation and the like and the advantages of the present invention will be retained.

I claim:

1. A method for separating a waxy mineral oil into several components including the steps of adding to the waxy mineral oil a methyl ethyl ketone toluol mixture consisting of a major portion of methyl ethyl ketone such that a solvent rich phase containing low viscosity index oil and a solvent lean phase containing high viscosity index oil will form at a temperature at least approximately 40° F. higher than a selected dewaxing temperature, chilling the resultant solution to the selected dewaxing temperature to cause it to form a solvent rich phase containing low viscosity index oil, a solvent lean phase containing high viscosity index oil and solidified wax, settling under quiescent conditions to separate the solvent rich phase from the remainder, recovering a first dewaxed oil phase from the solvent rich phase, adding a toluol-methyl ethyl ketone mixture consisting of a major portion of toluol to said remainder in an amount sufficient to wash substantially all of the oil from the solidified wax and subsequently separating in a second separation step a second dewaxed oil phase containing dewaxed oil of high viscosity index from a wax phase.

2. A method in accordance with claim 1 in which the second separation step is carried out by filtration.

3. A method in accordance with claim 1 in which the second separation step is carried out by settling.

4. A method for separating a waxy mineral oil into several components including the steps of adding to the waxy mineral oil a methyl ethyl ketone, toluol mixture

consisting of 75% to 100% methyl ethyl ketone and 25% to 0% of toluol such that the liquid will separate into a solvent rich phase containing low viscosity index oil and a solvent poor phase containing high viscosity index oil at a temperature no lower than 40° F., chilling the resultant solution to a temperature of approximately 0° F. to cause it to form a solvent rich phase containing low viscosity index oil, a solvent lean phase containing high viscosity index oil and wax crystals capable of settling to form from 25% to 50% of a clear layer in approximately 5 minutes, settling under quiescent conditions to separate a solvent rich phase and removing it from the remainder consisting of solvent poor phase and solidified wax in a first separation step, recovering a first dewaxed oil phase from the solvent rich phase, adding a toluol methyl ethyl ketone mixture consisting of 75% to 100% toluol and 25% to 0% of methyl ethyl ketone to the solvent poor phase and solidified wax in an amount sufficient to dissolve and wash substantially all of the oil from the wax and subsequently separating by filtration in a second separating step to form a solid wax phase and a second dewaxed oil filtrate phase containing a dewaxed oil of high viscosity index.

5. A method in accordance with claim 4 in which the first and second dewaxed oil phases are combined.

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