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SELECTIVE USE OF SOLVENT COMPONENTS IN SOLVENT DEWAXING

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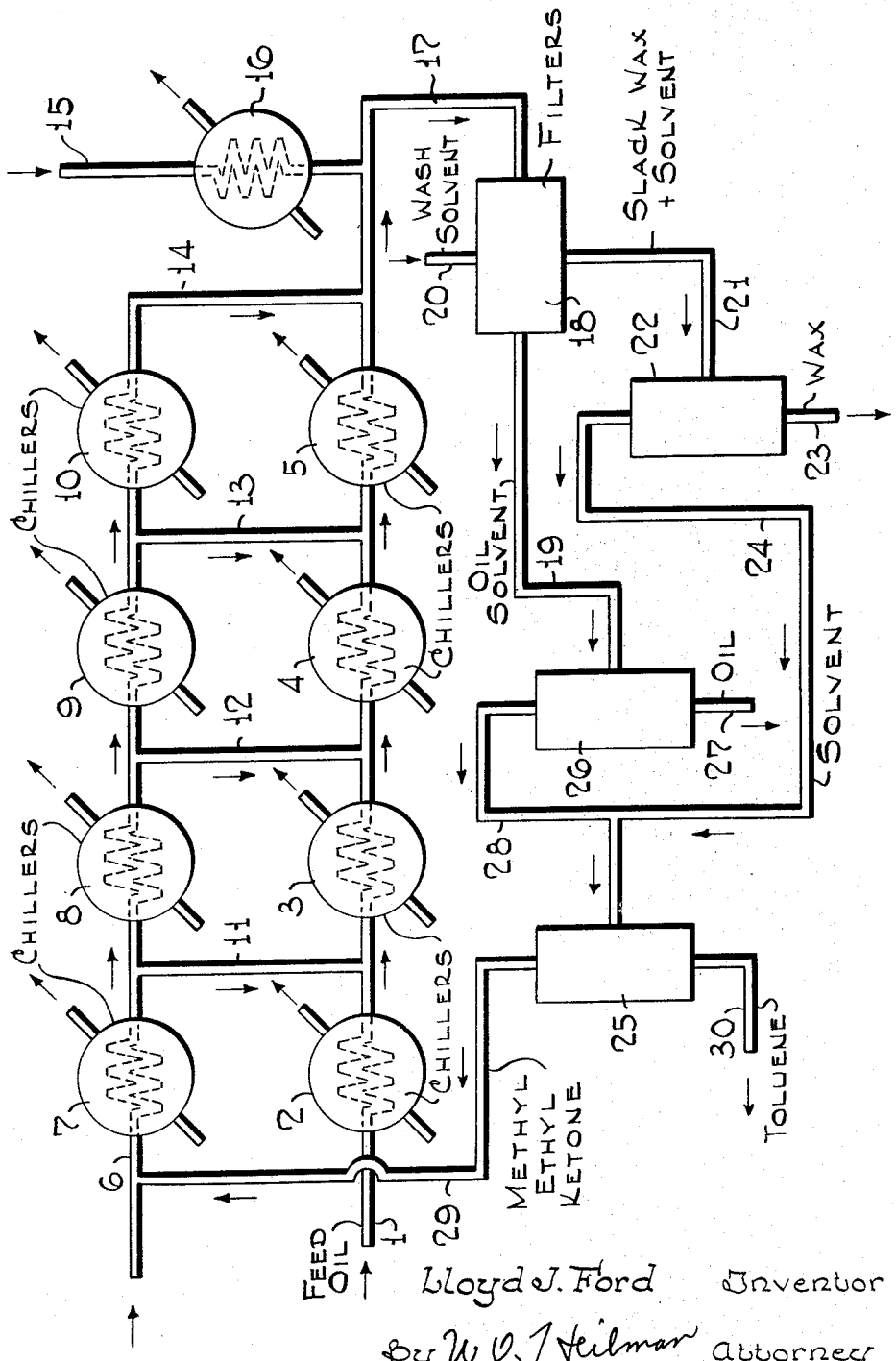


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# SELECTIVE USE OF SOLVENT COMPONENTS IN SOLVENT DEWAXING

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3 Claims. (Cl. 196—18)

The present invention is concerned with an improved process for the removal of waxy constituents from mineral oils containing the same. The invention is more particularly concerned with a dewaxing operation utilizing two solvents, one of which comprises an anti-wax solvent while the other of which comprises an oil solvent. The present invention is particularly concerned with the use of a solvent mixture in a dewaxing operation comprising a ketone and an aromatic as for example toluene in order to increase dewaxed oil yields and filtering rates. In accordance with the present invention the anti-wax solvent as for example the ketone is incrementally added to the waxy oil during the chilling cycle while the solvent having a solubility for oil is added to the chilled waxy oil in toto at the filtering temperature.

In the refining of hydrocarbon oils such as petroleum oils, it is known to segregate paraffin waxes from so-called paraffin distillates, waxy lubes and the like. The segregation of these waxes is secured by a number of processes. For example, it is known to chill the selected wax containing oil fraction in order to secure crystallization of the wax and then to filter or centrifuge the wax crystals from the oil. It is also known to use various dewaxing solvents such as liquid normally gaseous hydrocarbons, such as propane, as well as, other solvents such as methyl-ethyl ketone and the like. In these dewaxing operations, it is also known to use various filter aids and other agents in order to render the dewaxing and filtering operations more efficient. The wax segregated from the hydrocarbon oil, usually termed "slack wax" contains from about 10% to 40% of oil. The oil content and melting point range of this wax may be reduced if desirable by means of a distillation operation. The slack wax is then handled usually by sweating in a manner to reduce the oil content to less than about 4% by weight. This, "crude scale wax," generally has an oil content of about 2.5% by weight. In order to remove this oil from the wax and to produce a refined wax having an oil content below about .5%, usually below about .3% various procedures have been proposed and employed.

As pointed out, it is known to employ solvent mixtures wherein one solvent comprises an anti-wax solvent while the other comprises a solvent having a solubility for oil. A solvent mixture of this character for example comprises 60% by volume of methyl-ethyl ketone and 40% by volume of toluene. When utilizing a mixture of this character it has been the practice to add the mixture incrementally to the waxy distillate as it is being chilled. However, it has now been discovered that unexpected desirable results are secured providing the anti-wax solvent is added incrementally and the solvent having a solubility for oily constituents is added in toto at or near the filtering temperature. The process of the present invention may be more readily understood by reference to the drawing illustrating one embodiment of the same.

Referring specifically to the drawing, a waxy distillate is introduced into the system by means of feed line **1**. The waxy distillate flows serially through chilling zones



**2**, **3**, **4** and **5** wherein the temperature of the waxy distillate is progressively reduced. A typical operation is to introduce the feed oil into the initial chilling zone at a temperature of about 130° F.; to introduce the feed oil into the second chilling zone at a temperature of about 90° F.; to introduce the waxy distillate to the third chilling zone at a temperature of about 60° F.; to introduce the feed oil to the fourth chilling zone a temperature of about 25° F. and to chill the same in the fourth chilling zone to a filtering temperature in the range of from about −10 to +10° F. The number of the respective chilling zones as well as their arrangement may be varied appreciably and either direct or indirect chilling means utilized. For purposes of illustration it is assumed that the anti-wax solvent comprises methyl-ethyl ketone and that the aromatic solvent having a high solubility for oil comprises toluene. It is also assumed that 3½ volumes of total solvent mixture is utilized per volume of waxy oil being dewaxed. The solvent mixture comprises 60% by volume of methyl-ethyl ketone and 40% by volume of toluene.

The methyl-ethyl ketone is introduced into the system by means of line **6**, and at least a portion of the same passed serially through chilling zones **7**, **8**, **9** and **10**. The methyl-ethyl ketone in zone **7** is chilled to a temperature corresponding to the temperature of the waxy distillate removed from zone **2**. A portion of the methyl-ethyl ketone is added to the waxy distillate by means of line **11** while the remainder is chilled in zone **8** to a temperature approximating the temperature of the chilled distillate removed from zone **3**. A portion of the methyl-ethyl ketone removed from zone **8** is added to the waxy distillate removed from zone **3** by means of line **12**. The remainder of the methyl-ethyl ketone is further chilled in zone **9** to a temperature approximating the temperature of the distillate removed from zone **4**. Another portion of the methyl-ethyl ketone removed from zone **9** is added to the distillate removed from zone **4** by means of line **13**. The remainder of the methyl-ethyl ketone is chilled in zone **10** to a temperature approximating the temperature of the waxy distillate removed from zone **5** and added to the waxy distillate by means of line **14**.

The toluene in accordance with the present invention is introduced into the system by means of line **15**, chilled in zone **16** to a temperature approximating the temperature of the cold distillate removed from zone **5** and added to the distillate by means of line **17**. The entire mixture comprising oily constituents, crystallized wax constituents, toluene and methyl-ethyl ketone is held at this filtering temperature and passed to filtering zone **18** wherein the solid wax particles are segregated from the oily constituents by any suitable filtering or separation means. The filtering zone may comprise plate and frame presses, centrifuges or equivalent suitable equipment for the separation of the precipitated waxy constituents from the oily constituents. The oil and a portion of the solvent is removed from zone **18** by means of line **19** and the wax cake washed with a wash solvent introduced into filtering zone **18** by means of line **20**. Slack wax and solvent are removed from zone **18** by means of line **21** and passed to a distillation zone **22** wherein a separation is made between the waxy particles and the solvent. It is to be understood that other means of separating the solvent from the wax may be utilized if desirable. The wax substantially free of solvent is removed from separation zone **22** by means of line **23** and further refined as desired. The solvent mixture comprising methyl-ethyl ketone and toluene is removed overhead from zone **22** by means of line **24** and passed to distillation zone **25**.

The oil-solvent mixture removed from filtering zone **18** by means of line **19** is introduced into distillation zone **26** wherein a separation is made between the oily constit-

uents and the solvent mixture. The oil is removed from zone **26** by means of line **27** while the solvent mixture is removed overhead by means of line **28** and passed to distillation zone **25**. A separation is made in zone **25**, which may comprise a distillation zone, between methyl-ethyl ketone which is removed overhead by means of line **29** and the toluene which is removed as a bottoms stream by means of line **30**. It is to be understood that filtering zone **18**, distillation zones **22**, **25** and **26** may comprise any suitable number and arrangement of stages.

The methyl-ethyl ketone removed by means of line 29 is preferably recycled to the system and added in increments to fresh, waxy distillate while the toluene is recycled and added in toto at or near the filtering temperature by means of line **17**. Fresh methyl-ethyl ketone and toluene may be added as required.

The invention is broadly concerned with a dewaxing operation utilizing two solvents, one of which is characterized by being an anti-wax solvent, while the other is characterized by having a relatively high solubility for oily constituents. Solvents of the anti-wax characteristic, for example, comprise methyl-ethyl ketone, methyl-propyl ketone, acetone and the like, while solvents having a relatively high solubility for oily constituents comprise benzene, toluene and other aromatic solvents. The invention is specifically directed to a process wherein the anti-wax solvent is incrementally added to the waxy distillate as it is being chilled to a filtering temperature, while the aromatic solvent is added in toto at or near the filtering temperature. The method of adding the anti-wax solvent may be varied appreciably. It may be added in increments of from two to six and the quantity added as each increment varied appreciably. The amount added at each increment may be equal in volume, or a relatively small amount may be initially added and relatively larger amounts added in succeeding stages. For instance, a very satisfactory method of operation is to employ five chilling stages and to use about 350 volumes of a solvent mixture, comprising 60% by volume of methyl-ethyl ketone and 40% by volume of toluene, per 100 volumes of waxy oil being dewaxed. Under these conditions 30 volumes of methyl-ethyl ketone is added in the first chilling stage, 30 volumes in the second chilling stage, 60 volumes in the third chilling stage, 90 volumes in the fourth chilling stage and 140 volumes of toluene added at the filtering temperature.

The process of the present invention may be more fully appreciated by the following example illustrating the same:

*Example*

Various operations were conducted wherein a waxy feed having an API gravity of 32°, a 56 viscosity Saybolt seconds universal at 210, a Cleveland open cup flash of 440° F. was treated with a 3½ to 1 volumes of 60% methyl-ethyl ketone and 40% toluene by volume. In one series of operations the entire mixture was added in increments while in a second series of operations, the methyl-ethyl ketone was added in increments while the toluene was added in toto at the filtering temperature. The results of these operations are summarized as follows:

| | Operation I | Operation II |
|---|---|---|
| Methyl-ethyl ketone Added Increments | Yes | Yes. |
| Toluene Added Increment | Yes | No. |
| Filtration Rate, Gal./Hr./Sq. Ft. | 3.4 | 3.9. |
| Dewaxed Oil Yield | 78 | 89. |
| Dewaxed Oil Inspections: | | |
| Pour, ° F | 15 | 15. |
| Cloud, ° F | 18 | 18. |



From the above it is apparent that filtering rates are materially increased as well as dewaxed oil yields.

General operating conditions may be varied appreciably. For example, the concentration of the anti-wax solvent may vary from about 50% to 70% by volume while the concentration of the oil solvent may vary from 50% to 30% by volume. The amount of solvent mixture utilized may vary from about .5 to 8 volumes per volume of oil being dewaxed. Preferred ratios are from 3 to 5 volumes of solvent mixture per volume of oil being dewaxed. In accordance with the present invention, it is also preferred that the temperature of the oil solvent approximate the filtering temperature of the cold waxy distillate. In no instance should the temperature of the oil solvent added to the cold waxy distillate vary greater than about 10-20 degrees.

What is claimed is:

1. The process for the removal of waxy constituents from a a waxy oil containing same by the use of two different solvents, a ketone anti-wax solvent and an aromatic oil solvent, comprising the steps of chilling said waxy oil, adding sufficient ketone anti-wax solvent of substantially the same temperature to said waxy oil to precipitate only a portion of said waxy constituents, subsequently further chilling the resulting mixture of waxy oil, precipitated waxy constituents, and ketone anti-wax solvent to a lower temperature, subsequently adding additional ketone anti-wax solvent of substantially the same temperature as the further-chilled mixture to said further-chilled mixture to further precipitate said waxy constituents from said oil, adding to the resulting mixture subsequent to the addition of all the ketone anti-wax solvent to be introduced all of said aromatic oil solvent, said aromatic oil solvent being introduced at substantially the same temperature as the mixture to which it is added, and separating the precipitated waxy constituents at substantially the same temperature as said aromatic oil solvent from the resulting mixture of oil, aromatic oil solvent, and ketone anti-wax solvent.

2. The process according to claim 1 wherein said anti-wax solvent is methyl-ethyl ketone and wherein said oil solvent is toluene.

3. The process according to claim 2 wherein from 50–70 volumes of methyl-ethyl ketone are utilized per 30–50 volumes of toluene and wherein from 2–8 volumes of solvent mixture are utilized per volume of waxy oil being dewaxed.

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