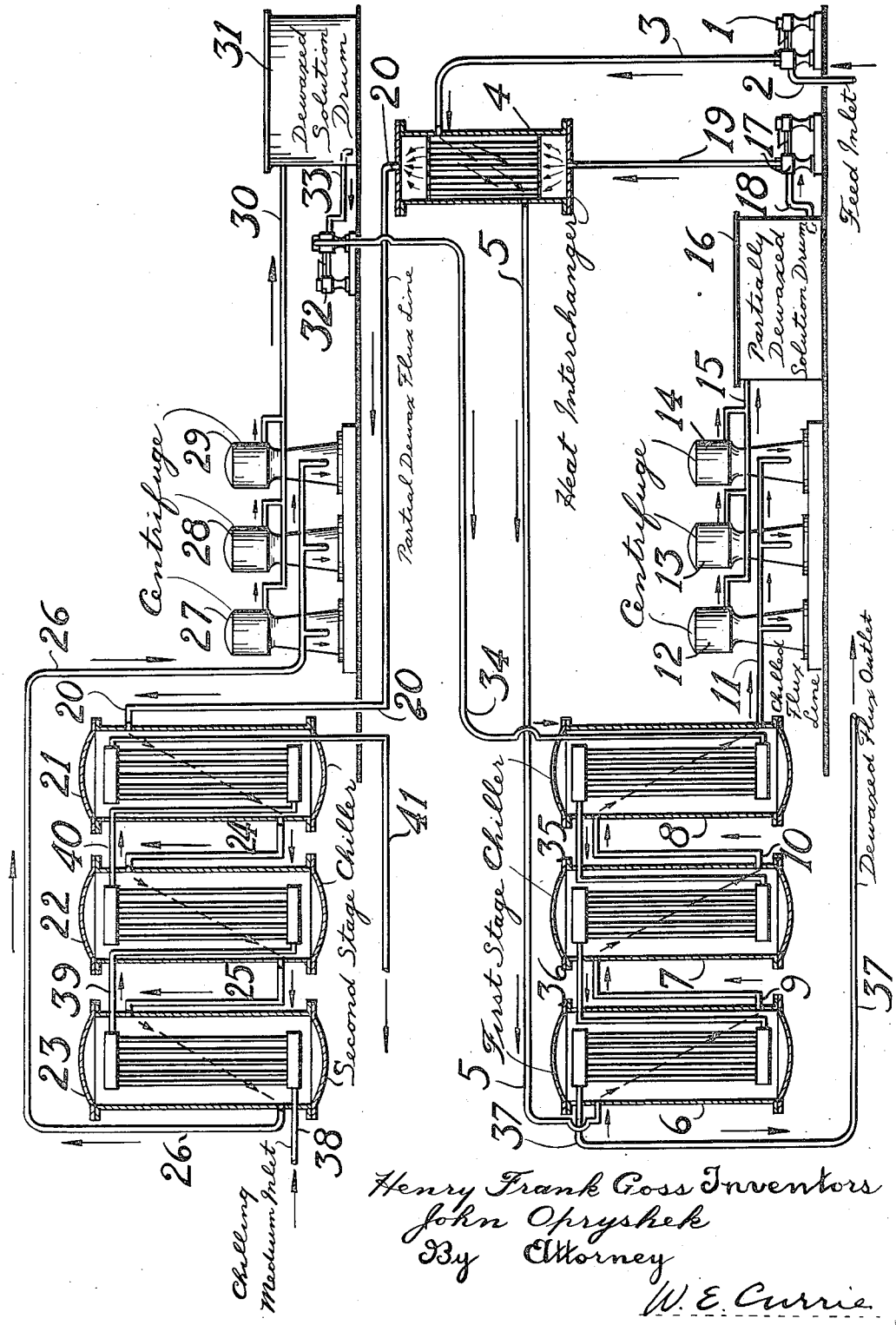
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DEWAXING HYDROCARBON OILS

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DEWAXING HYDROCARBON OILS

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2 Claims. (Cl. 196—18)

This invention relates to the dewaxing of hydrocarbon oils, especially petroleum oils, and more specifically relates to improvements in the dewaxing process.

The usual method of dewaxing hydrocarbon oils, such as the lubricating fractions of petroleum, comprises diluting the oil with several volumes of a solvent such as naphtha or lighter hydrocarbons, acetone, benzol, alcohol or the like, and then chilling the flux of oil and solvent to a temperature below the wax-separation point of the oil whereupon the wax is caused to precipitate and may be removed by cold-settling, centrifuging, filtering or other methods. The temperature to which the flux is chilled is determined by the pour point and cloud point desired in the final dewaxed oil.

We have found that with certain types of oils, especially those containing substantial amounts of the so-called amorphous waxes, removal of sufficient wax to lower the pour point to about 0° F. or below is difficult to accomplish by the usual methods of dewaxing. This invention therefore has for its principal object the provision of an improved method for substantially lowering the pour points of such oils.

The improvement comprises carrying out the dewaxing process in a series of stages, with alternate chilling and reheating of the flux between stages.

In the preferred embodiment of the invention the dewaxing is done in naphtha solution and the precipitated wax is removed between stages by centrifugal means. It should be understood, however, that the process is not limited to this diluent and method of removing precipitated wax, but that any other suitable means may be used.

The method of carrying out the process will be fully understood from the following description read with reference to the accompanying drawing which is a semi-diagrammatic view in sectional elevation of a suitable type of apparatus that may be used.

Referring to the drawing, numeral 1 designates a pump which draws the flux of oil and diluent at a temperature well above the wax separation point through line 2 and forces it through line 3 into a heat interchanging means 4 wherein it is partially cooled by interchange of heat with cold flux from the chillers. From the heat interchanger 4 the partially cooled flux flows through line 5 into the first stage chillers denoted by numerals 6, 7 and 8. The flux passes from chiller 6 to chiller 7 through line 9 and from chiller 7 to chiller 8 through line 10. The thoroughly chilled flux flows out of the last chiller 8 through line 11 and is passed into a series of centrifuges denoted by the numerals 12, 13 and 14 wherein as much as possible of the separated wax is removed.

The partially dewaxed flux leaves the centrifuges through line 15 and collects in drum 16 from which it is withdrawn by pump 17 through line 18 and forced through line 19 into heat interchanging means 4 wherein it serves partially to cool the entering warm flux and at the same time is reheated to a temperature at which finely divided particles of wax not removed in the centrifuges are redissolved. From the heat interchanging means 4 the reheated partially dewaxed flux flows through line 20 into a second series of chillers denoted by the numerals 21, 22, and 23 which are connected by means of lines 24 and 25. The rechilled flux passes out of the last chiller 23 through line 26 and is introduced into a second series of centrifuges denoted by the numerals 27, 28 and 29 wherein separated wax is again removed and from which the dewaxed flux is withdrawn through line 30 and collected in drum 31 from which it is withdrawn by pump 32 through line 33 and forced through line 34 into and through the first stage chillers where it serves as the cooling medium, flowing from chiller 8 to chiller 7 through line 35 and from chiller 7 to chiller 6 through line 36. From the last chiller the dewaxed flux is removed through line 37 and is then passed to suitable means (not shown) for removing the diluent from the dewaxed oil.

The chilling medium used in the second stage chillers may be introduced into chiller 23 through line 38 and will flow from one chiller to another through lines 39 and 40, leaving the last chiller through line 41. Any suitable chilling medium may be used, such as for example cold brine.

Although only two chilling stages have been shown in the drawing, it will be understood that as many additional chilling and centrifuging stages may be used as are found necessary to lower the pour point of the oil to the desired degree. It will also be understood that the temperature to which the chilled flux is reheated between stages may be suitably regulated by the provision of additional heating means (not shown), such as steam coils.

In the operation of our improved process the oil to be dewaxed is first diluted while maintained at a temperature well above the wax separation point, say about 100 to 150° F., with from 1 to 4 volumes of a suitable diluent such as naphtha. When thoroughly mixed the flux of oil and diluent is chilled to a temperature at which wax separates out, say 60° F. or below, depending upon the wax content and type of wax in the particular oil. The chilled flux is then centrifuged at this temperature in order to separate as much wax as possible from the flux. The partially dewaxed flux is then reheated to a temperature well above the wax separation point thereof in order to redissolve any small particles of wax that remain in the flux after centrifuging. The reheating may be conveniently effected by

interchange of heat with the warm entering flux. The reheated flux is again chilled, this time to a lower temperature than in the first chilling. The precipitated wax is again removed by centrifuging and the cycle of reheating and rechilling is repeated as many times as is found necessary to obtain a final oil of the desired low pour point. Chilling may be carried to −30° F., −40° F., −50° F., −60° F., and even lower in the successive steps and reheating may be carried to proportionally decreasing temperatures. The diluent naphtha may be removed from the final dewaxed flux by vaporization and the diluent so recovered may, it will be understood, be recycled through the process.

We have observed that when a flux of diluent and waxy oil is chilled in one step to a low temperature and the chilled flux then centrifuged, fine particles or crystals of wax remain in the flux and cannot be separated even by prolonged centrifuging. These fine particles cannot be removed by further chilling and centrifuging but appear to form a wax gel which favors the formation of additional fine gel-forming particles from the remainder of the wax. A further undesirable feature of the fine wax particles is that they decrease markedly the rate of heat transfer through the flux and this results in a loss of heat exchange efficiency.

The difficulties incident to the formation of the fine particles of wax are substantially avoided by the practice of the present improvement. Reheating of the flux after centrifuging causes the fine particles to redissolve and then upon rechilling the remaining wax is again precipitated in a form in which at least a substantial portion of it may be removed by centrifuging. As indicated above, the chilling may be carried to increasingly lower temperatures in each step. It is possible by this method to remove a greater proportion of the wax and to obtain oils of lower pour points and cloud points than can be accomplished by chilling to a very low temperature in a single step or in a series of steps without intermediate reheating.

The increased cost due to reheating and rechilling between stages is largely offset by the fact that a smaller quantity of diluent may be used than ordinarily, thus increasing the oil throughput of the centrifuges and decreasing the cost of refrigeration per gallon of oil, and also by the fact that much better heat transfer is obtained when the fine particles of wax are not present in the flux.

Various modifications of our improved process may be made without departing from the scope of the invention. Thus for example solvents other than naphtha may be used. Alcohols such as propyl and isopropyl alcohol, ketones such as methyl ketone, methyl ethyl ketone, esters such as secondary butyl acetate, liquefied hydrocarbons such as liquid ethane, propane and butane, and miscellaneous solvents such as benzol, toluol, acetone, methylene chloride, ethylene dichloride and so forth, or mixtures of any two or more of these may be used.

When using liquefied hydrocarbons such as propane, the chilling may be conveniently effected by auto-refrigeration, that is by direct vaporization of the propane from the flux by release of pressure.

Other methods of separating the precipitated wax may also be used such as those mentioned above. When working with oils containing substantial amounts of amorphous waxes, however, centrifuging is the preferred method of wax separation. Various filter aids such as finely divided clay, fuller's earth, silica and the like may be useful in facilitating the separation of the wax.

The following examples illustrate the application of our improved dewaxing method:

A wax-containing oil comprising a blend of oils obtained from Panhandle, Reagan and Salt Flat crudes and having the following characteristics:

Gravity, A. P. I.	26.8°
Pour point	115° F.
Flash point	515° F.
Saybolt viscosity at 100° F.	1130 seconds
Saybolt viscosity at 210° F.	84 seconds

is diluted with a naphtha having a gravity (A. P. I.) of 58°, so that the resulting blend comprises 30% oil and 70% naphtha. This flux is chilled to 50° F. and the chilled flux is then centrifuged at 50° F. in order to remove as much wax as possible. The partially dewaxed oil has a pour point of 80° F.

The flux is then reheated to a temperature of 90 to 100° F. and then rechilled to 10° F. and again centrifuged. The oil so obtained has a pour point of 55° F.

Again the remaining flux is reheated to 60° F. or higher and rechilled to −50° F. The oil now has a pour point of 0° F. It is reheated to 50° F. or higher and rechilled this time to −60° F. and the oil finally obtained has a pour point of −10° F.

The final dewaxed oil has the following inspection:

Gravity, A. P. I.	23.9°
Cloud point	0 to −10° F.
Pour point	−10°F.
Saybolt viscosity at 100° F.	1560 seconds
Saybolt viscosity at 210° F.	104 seconds

As another example, a treated wax-containing oil comprising a blend of oils obtained from Panhandle crude and having the following characteristics:

Gravity, A. P. I.	27.2°
Pour point	120° F.
Flash point	500° F.
Saybolt viscosity at 100° F.	725 seconds
Saybolt viscosity at 210° F.	70 seconds

is diluted with naphtha having a gravity of 57.2° A. P. I., so that the resulting blend comprises 28.5% oil and 71.5% naphtha. This flux is chilled to 30° F. and the chilled flux is then centrifuged at 30° F. in order to remove as much wax as possible. The partially dewaxed oil has a pour point of 65° F.

The flux is then heated to a temperature of 100° F. and then rechilled to −50° F. and again centrifuged. The dewaxed oil so obtained has the following inspection:

Gravity, A. P. I.	24.5°
Pour point	0° F.
Cloud point	0° F.
Saybolt viscosity at 100° F.	1044 seconds
Saybolt viscosity at 210° F.	82.6 seconds

In another experiment an untreated wax-containing oil comprising a blend of oils obtained

from Panhandle crude and having the following characteristics:

Gravity, A. P. I.	26.8°
Pour point	120° F.
Flash point	525° F.
Saybolt viscosity at 100° F.	850 seconds
Saybolt viscosity at 210° F.	73.2 seconds

is diluted with naphtha having a gravity of 57.2° A. P. I., so that the resulting blend comprises 33% oil and 67% naphtha. This flux is chilled to 30° F. and the chilled flux is then centrifuged at 30° F. in order to remove as much wax as possible. The partially dewaxed oil has a pour point of 65° F.

The flux is then heated to a temperature of 100° F. and then rechilled to —50° F. and again centrifuged. The dewaxed oil so obtained has the following inspection:

Gravity, A. P. I.	23.4°
Cloud point	0° F.
Pour point	0° F.
Saybolt viscosity at 100° F.	1337 seconds
Saybolt viscosity at 210°F.	91.6 seconds

This invention is not limited by any theory of the mechanism of the dewaxing nor by any details which have been given merely for purposes of illustration, but is limited only in and by the following claims in which we wish to claim all novelty inherent in the process.

We claim:

1. An improved process for dewaxing hydrocarbon oils which comprises diluting the oil with a suitable solvent, chilling the mixture to a temperature at which at least a portion of the wax is caused to precipitate, removing the precipitated wax by centrifugal means, reheating the partially dewaxed mixture to a temperature at which any unseparated wax is caused to redissolve, rechilling the mixture to a temperature at which wax is again caused to precipitate, removing this wax and recovering the dewaxed oil from the remaining mixture.

2. Process according to claim 1 in which the partially dewaxed mixture is rechilled to a temperature lower than that at which the mixture was chilled in the preceding step.

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