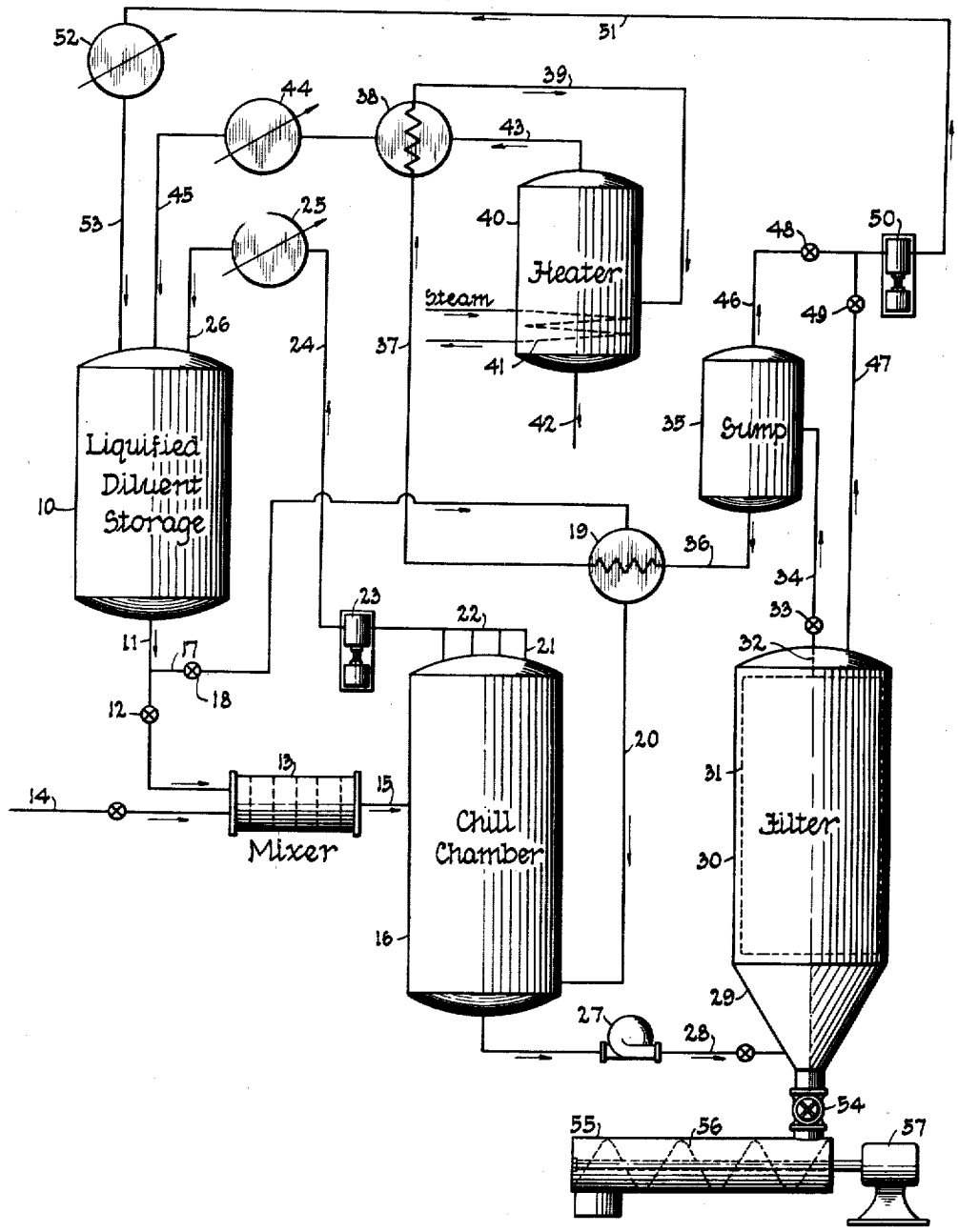
June 14, 1932. V. VOORHEES 1,862,874

SEPARATING WAX FROM OIL

Filed Oct. 26, 1931



Inventor

Vanderveer Voorhees

By Bruce K. Brown Atty.

Patented June 14, 1932 1,862,874

UNITED STATES PATENT OFFICE

VANDERVEER VOORHEES, OF HAMMOND, INDIANA, ASSIGNOR TO STANDARD OIL COMPANY, OF CHICAGO, ILLINOIS, A CORPORATION OF INDIANA

SEPARATING WAX FROM OIL

Application filed October 26, 1931. Serial No. 571,066.

This invention relates to a process for separating wax from oils and it pertains more particularly to the dewaxing of viscous petroleum lubricating oils to obtain desirable low pour point characteristics.

In the manufacture of petroleum lubricating oils it is necessary to remove substantially all of the wax in order that the oil may flow freely at low temperatures. This is usually accomplished by diluting a viscous wax-bearing oil with a light diluent, such as naphtha, chilling the mixture to about −20 to −40° F. to separate out the wax, and mechanically separating the wax from the diluted oil. The ease with which wax may be separated from oil depends on the form in which the wax is separated out, the solubility of the wax in the diluted oil, the viscosity and specific gravity of the diluted oil, etc. under the temperature and pressure conditions of the dewaxing apparatus. The object of my invention is to provide a diluent which will give oil mixtures of extremely low viscosity and low specific gravity, which will dissolve substantially no wax and which will bring about the solidification of wax in such a manner that it can be easily separated from the diluted oil. A further object is to provide a system for using this diluent in such a manner that the solidified wax will be completely thrown out of solution in a readily filterable form.

I have discovered that the normally gaseous alkyl and alkylene ethers are remarkably effective as antisolvents and diluents for the separation of wax from oil. These substances possess a low solubility for wax and their presence in a dewaxing mixture results in a substantially complete crystallization of wax in a form in which it can be readily removed from the oil. I prefer to use dimethyl ether which is a gas liquefying under pressure to a liquid having a low density and low viscosity. On reduction of pressure, the liquid boils at a low temperature, removing heat from substances with which it is in contact.

Examples of other ethers which I may use are methyl-ethyl ether and ethylene oxide, both readily liquefiable gases but having higher boiling points than dimethyl ether.

One of the novel features of my invention is the simultaneous refrigeration and antisolvent dewaxing of the oil by reducing pressure on the diluted wax-oil mixture, causing the evaporation of a portion of the low boiling ether. The rate of cooling can be easily regulated by controlling the pressure, which will, of course, have to be lower for the higher boiling ethers. When using dimethyl ether, pressures somewhat above atmospheric are suitable, while lower pressures may be used with higher boiling ethers. I may, if desired, use mixtures of the ethers with other solvents, such as the hydrocarbons, hexane, benzene, naphtha, etc. and particularly the liquefied, gaseous hydrocarbons, propane, butane, isobutane, or mixtures of propane and butane. The invention will be more clearly understood from the following detailed description of a preferred embodiment.

In the accompanying drawing, which forms a part of this specification, I have diagrammatically shown an elevational plan of my improved system.

The diluent storage tank 10 is preferably designed to withstand pressure and/or it may be insulated and provided with means for cooling the diluent which is stored therein. The diluent, for example, dimethyl ether, may be mixed with a light hydrocarbon preferably of about the same boiling point (e. g. propane), in which case the hydrocarbon will contribute to the refrigeration effect obtained. and the vapors of both hydrocarbon and ether will pass off together on evaporation.

The liquefied diluent is conducted from storage tank 10 through pipe 11 in amounts regulated by valve 12 into mixer 13, wherein it is mixed with the viscous wax-bearing lubricating oil introduced by pipe 14. The wax-bearing oil may be of any type, but in my preferred embodiment I have described a treated-wax-bearing, overheat lubricating cut having a viscosity of about 650 seconds Saybolt at 100° F. Such an oil may contain from 5–20% of wax.

The mixture of oil and diluent is introduced by pipe 15 into chill chamber 16 which

may be a large insulated drum. Instead of introducing all of the diluent with the oil I may bypass some or all of said diluent through pipe 17, valve 18, heat exchanger 19 and pipe 20 into the base of said chamber. By this expedient I am enabled to chill the diluent in the absence of wax so that there will be no accumulations of wax in the exchanger, the chilled diluent being introduced into the body of wax-bearing oil at the base of chill chamber 16. I prefer to mix about one-third of the diluent with the oil in mixer 13, fill the chill chamber about one-third full, then discontinue the introduction of the oil and introduce the remaining two-thirds of the diluent through pipe 17—20. Finally, I have about three to five parts of diluent to one part of waxbearing oil in the chill chamber.

By operating according to this plan the temperature of the mixture in the chill chamber may have been reduced from about 100° F. to about 40° F. by the bypassing of diluent through exchanger 19. This temperature reduction is preferably gradual (3° to 5° F. per minute) so that it will result in the formation of relatively large wax crystals. I continue the refrigeration by withdrawing diluent vapors through conduits 21, and manifold 22, compressing them by compressor 23 and returning them through pipe 24, condenser 25 and pipe 26 to the diluent storage tank 10. In this manner I effect self refrigeration, that is, I abstract heat from the liquid by the vaporation in situ of a portion of said liquid. The cooling from 40° F. to −10° F. may be at a more rapid rate, preferably about 8° to 10° F. per minute. Since the diluent itself has a tendency to throw wax out of solution, satisfactory pour points can be obtained at dewaxing temperatures of 0 to −10° F., the pour point being substantially the same as the dewaxing temperature.

The chilled slurry from chamber 16 is withdrawn through pipe 28 and pumped by pump 27 to the base 29 of filter 30 which is preferably of the vertical type wherein filter leaves 31 are suspended from a removable cover. Filtrate is withdrawn through conduit 32, valve 33 and pipe 34 to accumulation tank or sump 35, from which it may be withdrawn at desired intervals through pipe 36, exchanger 19, pipe 37, exchanger 38 and pipe 29, and introduced into boiler 40 which is heated by steam coils 41. The wax-free oil is withdrawn from the base of the boiler through pipe 42 and the diluent vapors are withdrawn through pipe 43, exchanger 38, condenser 44 and pipe 45 to diluent storage 10. Vapors from sump 35 and filter 30 are withdrawn through pipes 46 and 47 in amounts regulated by valves 48 and 49; these vapors are compressed in compressor 50 and returned through pipe 51 and condenser 52 to the storage tank 10.

When a cake has built up on the filter leaves, the liquid in the filter press is forced back into the chill chamber and the cake is dislodged from the leaves and withdrawn from the base of filter 30 through a large valve 54. It is then forced through conduit 55 by means of screw 56 driven by motor 57 into suitable apparatus for recovering diluent vapors and separating the wax from the entrained oil.

The particular feature of my invention is the use of a wax antisolvent as a refrigerant, the refrigeration being effected by the direct vaporization thereof. This brings about a saving in the cost of refrigeration which is augmented by the fact that the refrigeration need only be carried to about 0 to −10° F. to obtain the desired pour points, while heretofore it has been necessary to refrigerate with temperatures as low as −40° F. or even −60° F. The use of higher dewaxing temperatures also increases the rate of filtration and makes it possible to obtain a more complete separation of oil from wax on the filter cake.

While I have described in detail a preferred embodiment of my invention it is understood that I do not limit myself to the details hereinabove set forth except as defined by the following claims:

I claim:

1. The method of separating wax from viscous oils which comprises diluting said oil with a liquefied normally gaseous ether, chilling the diluted oil to a dewaxing temperature whereby the wax is thrown out of solution, and mechanically separating said wax from said diluted oil.

2. The process of claim 1 wherein the refrigeration is accomplished by direct vaporization of a portion of the diluent ether.

3. The process of claim 1 wherein a light hydrocarbon having about the same boiling point as the diluent is admixed with said diluent.

4. The process of dewaxing a viscous mineral oil comprising diluting said oil under pressure with a solvent selected from the class consisting of the normally gaseous alkyl and alkylene ethers, reducing the pressure on the solution, permitting a portion of the solvent to evaporate and thereby cooling the solution to a low temperature at which the wax is substantially all crystallized from the solution, separating the wax from the oil and solvent, and subsequently recovering the remaining solvent from the oil.

5. The process of dewaxing a mineral lubricating oil comprising diluting the oil under pressure with a diluent containing dimethyl ether, cooling the diluted oil by reducing pressure and evaporating a portion of dimethyl ether, thus causing the wax to crystallize from the diluted oil, and subsequently separating the wax from the oil.

6. The process of dewaxing a mineral lubricating oil comprising diluting the oil under pressure with a diluent containing methyl-ethyl ether, cooling the diluted oil by reducing pressure and evaporating a portion of the methyl-ethyl ether, thus causing the wax to crystallize from the diluted oil, and subsequently separating the wax from the oil.

7. The process of dewaxing a mineral lubricating oil comprising diluting the oil under pressure with a diluent containing ethylene oxide, cooling the diluted oil by reducing pressure and evaporating a portion of the ethylene oxide, thus causing the wax to crystallize from the diluted oil, and subsequently separating the wax from the oil.

In witness whereof I have affixed my signature.

VANDERVEER VOORHEES.

DISCLAIMER

1,862,874.—*Vanderveer Voorhees*, Hammond, Ind. SEPARATING WAX FROM OIL. Patent dated June 14, 1932. Disclaimer filed April 20, 1933, by the patentee, and the assignee, *Standard Oil Company* (*Indiana*).

Petitioners disclaim claims, 1, 2, 3, 4, and 5 of said patent to wit:

"1. The method of separating wax from viscous oils which comprises diluting said oil with a liquefied normally gaseous ether, chilling the diluted oil to a dewaxing temperature whereby the wax is thrown out of solution, and mechanically separating said wax from said diluted oil.

"2. The process of claim 1 wherein the refrigeration is accomplished by direct vaporization of a portion of the diluent ether.

"3. The process of claim 1 wherein a light hydrocarbon having about the same boiling point as the diluent is admixed with said diluent.

"4. The process of dewaxing a viscous mineral oil comprising diluting said oil under pressure with a solvent selected from the class consisting of the normally gaseous alkyl and alkylene ethers, reducing the pressure on the solution, permitting a portion of the solvent to evaporate and thereby cooling the solution to a low temperature at which the wax is substantially all crystallized from the solution, separating the wax from the oil and solvent, and subsequently recovering the remaining solvent from the oil.

"5. The process of dewaxing a mineral lubricating oil comprising diluting the oil under pressure with a diluent containing dimethyl ether, cooling the diluted oil by reducing pressure and evaporating a portion of dimethyl ether, thus causing the wax to crystallize from the diluted oil, and subsequently separating the wax from the oil."

[*Official Gazette May 16, 1933.*]

6. The process of dewaxing a mineral lubricating oil comprising diluting the oil under pressure with a diluent containing methyl-ethyl ether, cooling the diluted oil by reducing pressure and evaporating a portion of the methyl-ethyl ether, thus causing the wax to crystallize from the diluted oil, and subsequently separating the wax from the oil.

7. The process of dewaxing a mineral lubricating oil comprising diluting the oil under pressure with a diluent containing ethylene oxide, cooling the diluted oil by reducing pressure and evaporating a portion of the ethylene oxide, thus causing the wax to crystallize from the diluted oil, and subsequently separating the wax from the oil.

In witness whereof I have affixed my signature.

VANDERVEER VOORHEES.

DISCLAIMER

1,862,874.—*Vanderveer Voorhees*, Hammond, Ind. SEPARATING WAX FROM OIL. Patent dated June 14, 1932. Disclaimer filed April 20, 1933, by the patentee, and the assignee, *Standard Oil Company* (*Indiana*).

Petitioners disclaim claims, 1, 2, 3, 4, and 5 of said patent to wit:

"1. The method of separating wax from viscous oils which comprises diluting said oil with a liquefied normally gaseous ether, chilling the diluted oil to a dewaxing temperature whereby the wax is thrown out of solution, and mechanically separating said wax from said diluted oil.

"2. The process of claim 1 wherein the refrigeration is accomplished by direct vaporization of a portion of the diluent ether.

"3. The process of claim 1 wherein a light hydrocarbon having about the same boiling point as the diluent is admixed with said diluent.

"4. The process of dewaxing a viscous mineral oil comprising diluting said oil under pressure with a solvent selected from the class consisting of the normally gaseous alkyl and alkylene ethers, reducing the pressure on the solution, permitting a portion of the solvent to evaporate and thereby cooling the solution to a low temperature at which the wax is substantially all crystallized from the solution, separating the wax from the oil and solvent, and subsequently recovering the remaining solvent from the oil.

"5. The process of dewaxing a mineral lubricating oil comprising diluting the oil under pressure with a diluent containing dimethyl ether, cooling the diluted oil by reducing pressure and evaporating a portion of dimethyl ether, thus causing the wax to crystallize from the diluted oil, and subsequently separating the wax from the oil."

[*Official Gazette May 16, 1933.*]