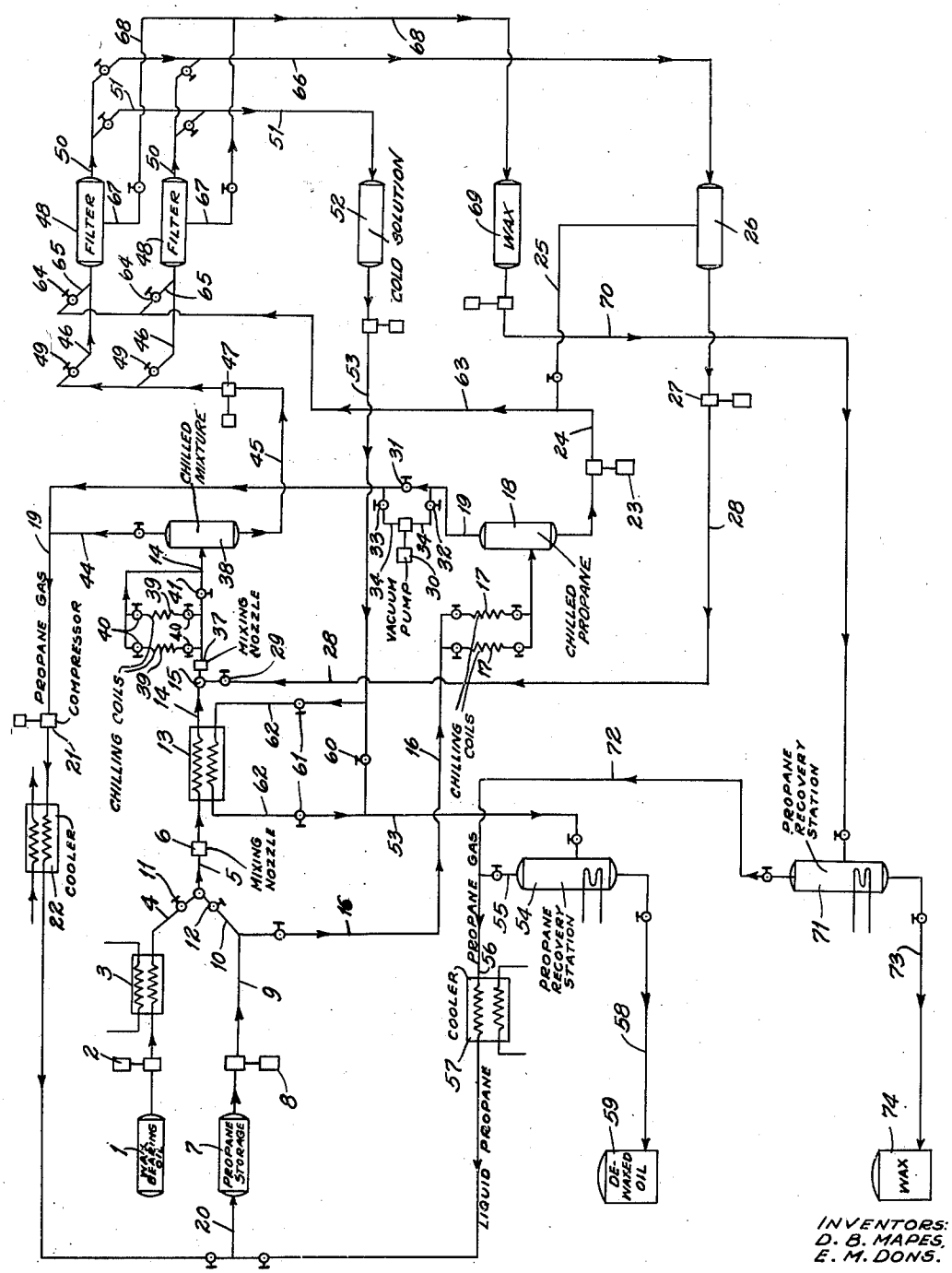
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PROCESS OF DEWAXING OILS

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## PROCESS OF DEWAXING OILS

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This invention relates to processes of dewaxing oils, and more particularly to the dewaxing of oils wherein a solvent, or other fluid, is employed to chill the oil. This application is a continuation in part of an application for patent filed by us on May 3, 1934, Serial No. 723,662.

In the usual methods of chilling wax-containing oils to precipitate wax, it has been generally recognized that the condtions of chilling must be carefully controlled, otherwise wax will be precipitated in such a form that it cannot be readily separated from the oil.

Prior to our inventions in this art, it has been generally believed that any rapid chilling or "shock chilling" of oil solutions would produce a wax structure that could not be separated from the oil. In the usual dewaxing operations it has, therefore, been considered highly important to very slowly chill wax-containing oil solutions, so that the wax will be precipitated in a form that can be readily removed from the solution.

However, the present invention includes a new process wherein the rate of chilling is not the critical factor, and under certain conditions such a solution can be suddenly chilled to very low temperatures, with the result of quickly providing a chilled solution from which the precipitated wax can be readily separated.

Our process is suitable for dewaxing oils with normally gaseous hydrocarbons, such as propane. However, other solvents and mixtures of solvents, including naphtha, naphtha and propane, may be advantageously employed to obtain some of the results of our invention.

The method of chilling the solution of oil and propane is very important, for if the solution is not properly chilled the wax cannot be successfully filtered from the solution.

Due to the results obtained from improper chilling, it has been thought that the batch method of chilling was the only manner in which the required chilling could be obtained. A batch operation provides satisfactory conditons for chilling the solution of oil and propane, as the propane may be very gradually released from a large volume of the solution at the desired low rate to precipitate the wax in the form desired for filtering.

However, in a large commercial dewaxing plant, such batch operations have serious disadvantages. Large vessels of alloy steel subjected to a high pressure must be repeatedly warmed and then chilled to about —40° F., which is an uneconomical and inefficient procedure. Moreover, the hazards involved in such rapid temperature changes in high pressure vessels, necessitates a very expensive construction. In addition to the foregoing disadvantages the volume of propane required is large and the production slow.

An object of our invention, therefore, is to provide a continuous process for dewaxing an oil with propane wherein a constantly flowing stream is continuously chilled under conditions that result in a chilled solution from which the wax can readily be removed by filtration.

Another object of this invention is to provide a process and apparatus wherein the temperature at any point in the system will be practically constant so as to avoid the strain of frequent and rapid temperature changes.

A further object is to provide a simple and relatively inexpensive process for dewaxing an oil with propane while obtaining a large production of dewaxed oil with a relatively small quantity of propane in the system.

A still further object of this invention is to provide a process for the rapid chilling of wax-containing oils, so as to quickly precipitate wax in a considerable less time than is usually required.

With the foregoing and other objects in view, the invention comprises the novel method hereinafter more specifically described and shown in the accompanying drawing, which illustrates one form of the invention. However, it is to be understood that the invention comprehends changes, variations and modifications which come within the scope of the claims hereunto appended.

As an illustration of the prior methods, we will refer to the well known use of propane solutions wherein the propane is slowly evaporated to very gradually chill the solution to the desired low temperature, the object being to prevent rapid evaporation which would result in rapid chilling, and produce an undesirable wax structure that could not be satisfactorily separated from the solution. This is true of the methods of using propane prior to our inventions, and such methods were limited to relatively slow and expensive batch operations.

Nevertheless, in one form of our invention, we employ propane under conditions which permit the wax-containing oil to be suddenly chilled while it is quickly flowing, in a continuous stream, from a source of supply to a separating station where the congealed wax is removed from the continuous stream. In other words, by overcoming the conditions requiring slow chilling, and batch operations, we have produced a continuous system wherein the wax can be rapidly con-

gealed in a continuous stream, and then readily removed from the oil.

However, it is to be understood that the scope of the present invention extends to combinations of steps which may, if desired, include gradual chilling, and even batch operations in parts of system conforming to the invention.

The drawing is a diagrammatical view of a system embodying the features of this invention.

We have found that wax having the desired structure can be very effectively precipitated from a wax-containing oil, or a solution of oil and solvent, by a very sudden chilling of the oil, but if the cooling agent is united with the oil during the rapid chilling operation, it should not be subjected to a correspondingly rapid expansion.

In the preferred form of our invention, we accomplish the sudden chilling of the oil by introducing a stream of cold solvent directly into a stream of relatively warm oil and solvent to almost instantly chill the oil and precipitate wax. If the oil solution has not been subjected to a correspondingly rapid expansion during the chilling operation, the wax will have a structure which permits it to be as freely filtered from the oil as wax precipitated by a very gradual chilling of an oil.

The oil to be dewaxed may be chilled to any desired temperature by a regulation of the temperature and proportion of the cold liquid to be mixed with the oil. Moreover, our invention may be advantageously employed in the usual dewaxing processes as a preliminary step to precipitate a portion of the wax in the oil. An additional quantity of wax may, thereafter be precipitated by a gradual chilling of the oil and solvent. This preliminary treatment is especially advantageous when it is not desirable to chill the solvent to a temperature low enough to completely dewax the oil.

As an illustration of our invention, we have shown a system wherein a normally gaseous hydrocarbon, such as propane, may be employed as the solvent. However, it is understood that other normally gaseous hydrocarbon and normally liquid solvents, as well as mixtures of various solvents, may be employed to obtain some of the advantages of our invention.

Undewaxed oil is continuously withdrawn from a storage tank 1 by a pump 2 and passed through a heater 3, and conductors 4 and 5 into a mixing nozzle 6. The oil is preferably warmed in heater 3 to a temperature at which it is freely soluble in liquid propane, such as 175° F.

Liquid propane is continuously withdrawn from storage tank 7 by a pump 8 and forced under a pressure sufficient to maintain the propane in a liquid condition through conductors 9, 10 and 5 to the mixing nozzle 6 where it is intimately mixed with the warm oil from storage tank 1. Valves 11 and 12 in the respective conductors 4 and 10 may be regulated to obtain the desired ratio of oil to propane, such as about one part oil to 1½ parts propane. The solution of oil and propane may then be passed through a heat exchanger 13 where it is cooled to about 60° F., or to a temperature at which a cloud of wax begins to appear in the oil.

The solution of oil and propane passing from the heat exchanger 13 through conductor 14 is suddenly chilled to quickly precipitate wax by introducing a cold stream of propane into the solution of oil and propane at a mixing head 15.

To provide for the rapid chilling of the stream of solution at mixing head 15, propane from storage tank 7 may be passed through conductors 9 and 16 to chilling coils 17 where the pressure on the propane is released to evaporate a portion of the propane and chill the remaining propane to a very low temperature. The chilled propane upon leaving the chilling coils 17 is transmitted to a receiver 18. The gaseous propane in receiver 18 is returned to the storage tank through conductors 19 and 20, and is preferably condensed by compressor 21 and condenser 22 so that the propane is returned in a liquid condition to storage tank 7.

The chilled liquid propane in receiver 18 may be forced by pump 23 through conductors 24 and 25 to a reservoir 26. From the reservoir 26 the chilled liquid propane is forced by pump 27 through a conductor 28 into the stream of solution at the mixing head 15 to rapidly chill the solution and quickly precipitate wax. The conductor 28 may be provided with a valve 29 to provide for the regulation of the quantity of chilled propane introduced into the stream of solution.

If it is desired to chill the oil solution to the desired low temperature to precipitate wax by the injection of extremely cold propane alone, the propane is preferably chilled by reducing the pressure thereon in the receiver 18 to a pressure less than atmospheric pressure. To provide for the reduction in pressure on the propane in receiver 18, we have shown a vacuum pump 30, which may be operatively connected to the receiver 18 by closing valve 31 in the conductor 19 and opening valves 32 and 33 in conductors 34. By the operation of vacuum pump 30, the propane may be chilled to a very low temperature, such as from —60° to —90° F., depending upon the reduction in pressure.

By chilling the propane to about —90° F. and introducing the propane into the oil solution passing through conductor 14 at the rate of 2.6 parts chilled propane to one part solution, the mixing of the two streams will result in a solution having a temperature of about —40° F. In other words, the temperature of the oil solution may be suddenly lowered from about 60° F. to —40° F. to suddenly precipitate wax contained in the oil. The resultant congealed wax will have a structure that can be readily filtered from the oil solution.

The resultant chilled mixture, after passing through mixing head 15 and mixing nozzle 37, may be transmitted to a reservoir 38 prior to filtering the wax from the oil.

If desired, the pressure on the propane passing from the storage tank 7 through chilling coils 17 and into receiver 18 may be reduced to atmospheric pressure, and the operation of the vacuum pump 30 eliminated by closing valves 32 and 33 and opening valve 31. In this case, the temperature of the chilled propane, which may be from —40° F. to —44° F., will be substantially warmer than when the propane was evaporated under a pressure less than atmospheric pressure.

The introduction of propane at a temperature of —40° F. to —44° F. into the oil solution at 60° F. will result in a temperature of about 10° F. If it is desired to chill the resultant chilled solution to a lower temperature to precipitate an additional quantity of wax, the solution after passing from mixing head 15 and mixing nozzle 37, may be passed through chilling coils 39, by opening valves 40 at the inlets and outlets of

either or both of the coils 39 and closing the valve 41 in the conductor 14.

These chilling coils will provide a gradual decrease in pressure on the solution, whereby a portion of the propane is evaporated to effect a gradual reduction in the temperature as the stream passes through the coil. This feature of the method is disclosed and claimed in the patent application filed by us on May 3, 1934, Serial No. 723,662.

The solution passing from the coils 39 is transmitted to the reservoir 38. The reservoir 38 is provided with a propane vapor outlet pipe 44 leading to return conductor 19 to provide for a return of the propane to the storage tank 7.

The cold solution of propane and oil in reservoir 38, which may be obtained either by injecting cold propane alone into the oil solution, or by injecting cold propane and thereafter evaporating a portion of the propane in the solution, is forced by pump 47 through conductor 45 and one of the branch conductors 46, into one of the filters 48. The branch conductors 46 are provided with valves 49 so that the solution may be diverted from one filter to the other when the filter in service becomes clogged with wax. The cold wax-free solution flows from filters 48 through conductors 50 and 51 to a receiver 52, from which it is pumped through a conductor 53 to a solvent recovery station 54. At the solvent recovery station 54 propane may be recovered by any suitable method, such as by heating the solution, reducing the pressure on the heated solution, and stripping with live steam. The recovered propane is withdrawn through a conductor 55 leading to a conductor 56 and, after being condensed in cooler 57, is returned to the propane storage tank 7. The dewaxed oil passes from the solvent recovery station 54 through a conductor 58 to a storage tank 59.

The chilled oil passing from the receiver 52 may be advantageously used as a chilling medium in the heat exchanger 13. By closing a valve 60 in the conductor 53 and opening valves 61 in a conductor 62 passing through heat exchanger 13, cold oil will be passed through said heat exchanger 13 before it reaches the solvent recovery station 54.

As previously stated, the cold solution of propane and oil in reservoir 38 may be transmitted to either of filters 48, so that when the filter in service becomes clogged with wax the other filter is available to prevent an interruption of the continuous operation of the system.

When one of the filters becomes clogged, the wax therein is preferably treated with propane to wash the oil from the wax cake in the filter.

To provide for the removal of the oil from the wax in the filter out of service, cold liquid propane from receiver 18 may be pumped through conductors 24 and 63, and by regulating valves 64 in the branch conductors 65, the propane may be passed into the selected filter to wash the oil from the wax cake.

The resultant cold filter-wash passes from either of the filters 48 through conductors 50 and 66 to the reservoir 26, from which it may be forced through conductors 28 to the fresh solution flowing through the conductor 14 to aid in chilling the oil.

The wax, and any propane contained therein, may be transmitted from either of the filters 48 by conductors 67 and 68 to a receiver 69. From the receiver 69 wax may be transmitted through a conductor 70 through a propane recovery station 71 similar to the propane recovery station 54. The recovered propane is returned to the storage tank through conductors 72, 56 and 20. The wax passes from the solvent recovery station 71 through a conductor 73 to a wax storage tank 74.

While we have given specific illustrations of one process, it is understood that numerous changes and modifications may be made without departing from the spirit of this invention as set forth by the claims.

It is also understood that the term propane does not necessarily imply a pure propane, as propane is employed to cover a mixture of propane and other hydrocarbons. For example, a mixture comprising of about 80% propane and about 20% ethane, and butanes is quite satisfactory for use in carrying out this invention.

As previously stated, propane is a normally gaseous hydrocarbon, and in the preferred form of this invention we prefer to use propane for treating the oil. However, examples of other normally gaseous hydrocarbons include ethane, propylene, and isobutane.

This invention provides a very simple, inexpensive and efficient process for the continuous dewaxing of an oil with a solvent, wherein the solvent alone may be employed to quickly chill relatively warm oil to a very low temperature, thereby quickly precipitating wax having a structure which is freely removable from the oil.

The solvent may be chilled prior to the treatment of the oil in any desired manner, such as by evaporating the solvent when a normally gaseous hydrocarbon is employed, or any other suitable method may be employed to suddenly chill and rapidly congeal the wax.

The hazard of high pressure vessels, except for storage purposes, is eliminated in carrying out our process. The equipment employed to carry out this process is not subjected to the strain of frequent temperature changes, as the temperature at any given point in the system is practically constant after the system is in operation.

The total volume of solvent in the system is relatively small compared to the capacity of the plant, and a relatively large quantity of dewaxed oil can be quickly obtained by the sudden chilling of oil in a constant stream passing from a source of supply to the filter. Moreover, the cost of the plant is relatively inexpensive and many dangers of dewaxing plants are materially reduced.

We claim:

1. The process of dewaxing oils which comprises dissolving a wax-containing oil in a liquefied normally gaseous hydrocarbon, cooling the solution to approximately the temperature at which a cloud of wax appears, suddenly cooling the resultant solution to quickly precipitate wax therein by introducing a stream of chilled normally gaseous hydrocarbon liquid into a stream of said solution, thereafter precipitating additional wax from the cooled oil while forcing a stream of the resultant material through an elongated passageway to provide a gradual decrease in pressure on the flowing stream and a gradual evaporation of a portion of the normally gaseous hydrocarbon, and separating the precipitated wax from the oil.

2. The process of dewaxing oils which comprises dissolving a wax-containing oil in a liquefied normally gaseous hydrocarbon, cooling the solution to a temperature above the desired de-

waxing temperature, suddenly cooling the resultant solution to quickly precipitate wax therein at temperatures substantially below 60° F. by introducing a stream of chilled normally gaseous hydrocarbon liquid into a stream of said solution, thereafter precipitating additional wax from the cooled oil while forcing a stream of the resultant material through an elongated passageway to provide a gradual decrease in pressure on the flowing stream and a gradual evaporation of a portion of the normally gaseous hydrocarbon, and separating the precipitated wax from the oil.

3. The continuous process of dewaxing oils which comprises dissolving a stream of wax-containing oil in a stream of propane while subjecting the solution to a pressure sufficient to maintain the propane in a substantially liquid condition, regulating the flow of said streams to produce a solution wherein the quantity of propane is larger than the quantity of said oil, cooling the solution to a temperature above the desired dewaxing temperature, suddenly cooling the resultant solution to quickly precipitate wax therein at temperatures substantially below 60° F. by introducing a stream of chilled propane into a stream of said solution, thereafter precipitating additional wax from the cooled oil while forcing a stream of the resultant material through an elongated passageway to provide a gradual decrease in pressure on the flowing stream and a gradual evaporation of a portion of the propane, and separating the precipitated wax from the oil.

4. The continuous process of dewaxing oils which comprises dissolving a stream of wax-containing oil in a stream of propane while subjecting the solution to a pressure sufficient to maintain the propane in a substantially liquid condition, regulating the flow of said streams to produce a solution having about one part of said oil to about one and one-half parts propane, cooling the solution to a temperature above the desired dewaxing temperature, suddenly cooling the resultant solution to quickly precipitate wax therein at temperatures between 60° F. and 10° F. by introducing a stream of chilled propane into a stream of said solution, thereafter precipitating additional wax from the cooled oil while forcing a stream of the resultant material through an elongated passageway to provide a gradual decrease in pressure on the flowing stream and a gradual evaporation of a portion of the propane, and separating the precipitated wax from the oil.

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