

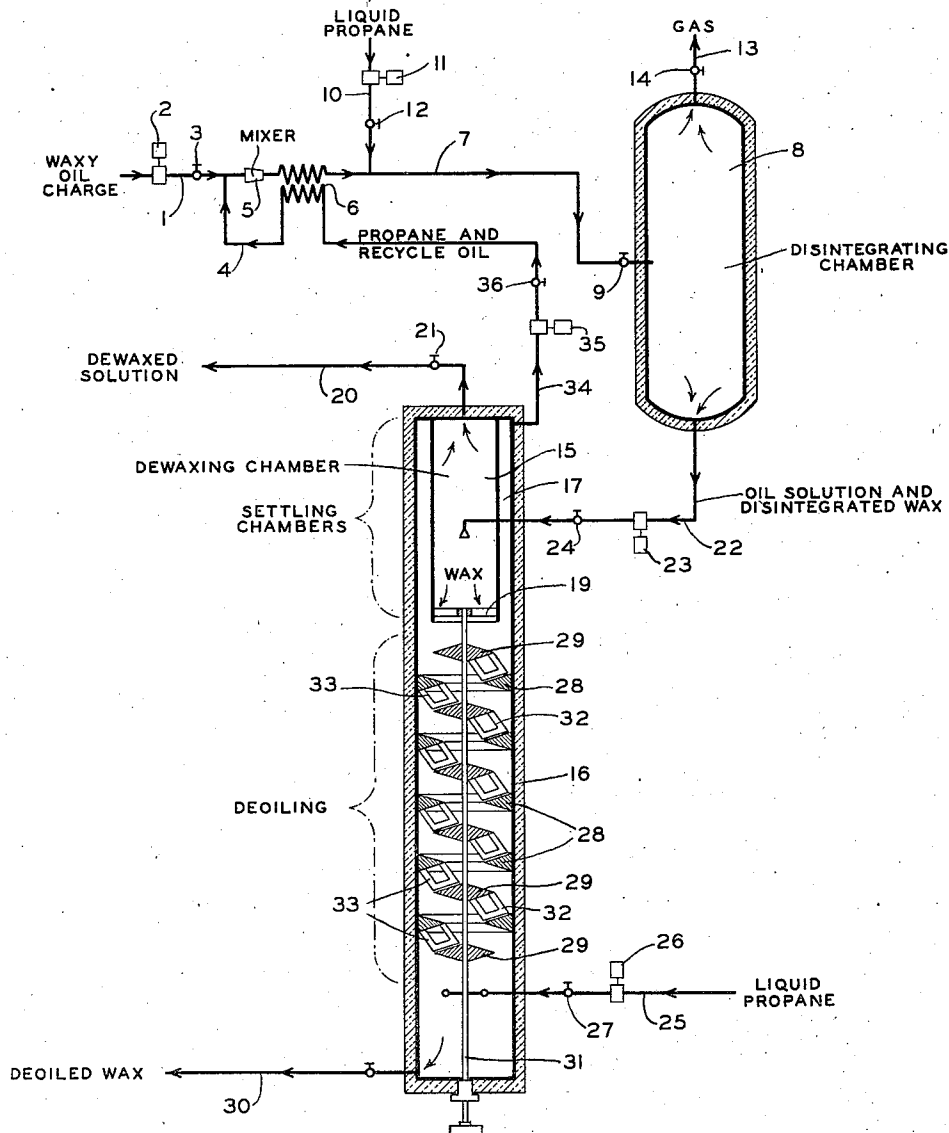
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PROCESS OF USING NORMALLY GASEOUS SOLVENTS TO SEPARATE WAX AND OIL

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PROCESS OF USING NORMALLY GASEOUS SOLVENTS TO SEPARATE WAX AND OIL

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This invention relates to processes of using normally gaseous solvents to separate wax and oil. The invention may be employed in separating wax and oil found in various kinds of materials including mineral oils, petrolatum and slack wax. It can be conveniently used in dewaxing petroleum lubricating oils to produce low pour point lubricants.

The main object of the invention is to produce a relatively simple and inexpensive method of using normally gaseous dewaxing solvents lacking several serious objections found in prior processes. The commercial development of dewaxing systems has passed through a series of stages, which eventually led to the modern dewaxing systems wherein extremely large filters are employed to separate wax from diluted oil.

Nevertheless, the highly developed conventional filtering processes now in general use are complex systems, involving considerable trouble and expense, and lacking the desired efficiency in the separation of wax from oil. In commercial practice, clusters of wax crystals are deposited on the filters with portions of the oil solution trapped or occluded in the wax clusters. The filtration does not remove this occluded oil solution from the wax clusters, and additional portions of the solution are trapped between the clusters on the filter.

A primary object of the invention is to advance this art by producing a very simple and inexpensive commercial system, lacking inherent defects, or objections, found in the dewaxing systems which have been generally adopted in this industry.

A specific object of the present invention is to produce an entirely feasible dewaxing system wherein normally gaseous dewaxing solvents are deliberately employed to forcibly disintegrate the wax. We realize that experts in this art have regarded even a slight or incidental disintegration of the wax as a nuisance or objectionable condition to be carefully avoided in the separation of wax and oil. However, a study of the plan herein set forth will show that we have established novel conditions wherein this radical departure from the usual practice is employed to obtain substantial advantages.

Instead of exercising precautions to avoid a possibility of slight disintegration, we have created cooperative conditions wherein rather severe disintegration of the wax is an outstanding factor tending to promote separation of the wax and oil.

For example, propane is a very popular normally gaseous solvent having special advantages for dewaxing, including the intense chilling which occurs when propane is evaporated in the oil solution. However, in commercial practice special precautions are exercised to avoid a sud-

den expansion which would rupture the precipitated wax clusters, or otherwise result in disintegration of the wax. This usual commercial practice has resulted in so called "critical" rates of chilling, requiring gradual evaporation of propane, intended to avoid "shock chilling" which would rupture the wax clusters, or otherwise interfere with the usual methods of separating the wax from the propane solution. In this popular commercial procedure, the free oil solution is transmitted through a filter, while substantial portions of the solution are trapped, or occluded, in the wax cake deposited on the filter.

To overcome these problems, we will show how the wax can be deliberately subjected to a forcible disintegrating operation, which liberates practically all of the oil solution from the wax. This may be regarded as a desirable preliminary step in the new sequence of operations wherein the departure from conventional theories is employed to improve the art of using normally gaseous dewaxing solvents.

A special advantage is gained by suddenly evaporating portions of the normally gaseous material in the precipitated wax, as this will result in a sudden explosive force which forcibly disintegrates the wax, thereby positively releasing oil solution which is usually trapped in the wax. The liberated oil solution will then be free to separate from the wax in the dewaxing or deoiling operation. This is a substantial advantage over the usual commercial practice, wherein trapped or occluded oil is inevitably retained within and between wax clusters deposited on the filters.

A separate and distinct object of the present invention is to obtain advantages of using a normally gaseous solvent, including the advantage of evaporating portions of the solvent to produce the desired degree of chilling, and at the same time avoid the usual trouble, expense and inefficiency involved in the ordinary filtering plants. This phase of the invention may be advantageously employed in the sequence of operations which include forcible disintegration of the wax, but it has special advantages which may be gained without subjecting the wax to said forcible disintegration.

Briefly stated, the liquefied normally gaseous solvent may be mixed with the wax-containing oil under a pressure high enough to retain said solvent in a liquid condition. The pressure is thereafter reduced, either with or without forcibly disintegrating the wax, so as to evaporate portions of the solvent and thereby chill the solution to precipitate wax therein. However, instead of filtering the resultant mixture of wax and oil solution, this mixture is preferably transmitted into a settling chamber. A rising stream of dewaxed solution may be discharged from an

upper portion of the settling chamber, thereby dewaxing a major portion of the oil solution. A descending stream of wax with portions of the oil solution is preferably discharged from a lower portion of the settling chamber to a counterflow deoiling system, where the wax is subjected to a deoiling operation and then discharged from the system.

The deoiling operation may be performed by transmitting a rising stream of liquefied normally gaseous solvent through a descending stream of wax, so as to dissolve oil solution carried by the wax, and we will hereafter show how a stream of the used deoiling solvent, with the oil solution dissolved therein, may be transmitted to the wax-containing charging stock, so as to recycle the dissolved oil from the deoiling system, and at the same time utilize the deoiling solvent in forming the dewaxing solution.

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

The accompanying drawing is a diagrammatical view of a system which may be employed to carry out one form of the invention.

A continuous regulated stream of the wax-bearing charging stock, for example, a lubricating oil stock, enters the system through a pipe 1 which may be provided with a pump 2 and a regulating valve 3. A continuous stream of liquefied normally gaseous dewaxing solvent, from sources to be hereafter described, is conducted through a pipe 4 to the oil supply pipe 1. The continuous streams of oil and dewaxing solvent are united in the pipe 1 and transmitted through a mixing nozzle 5 where the oil and solvent are mixed at a temperature high enough to form the desired solution.

The continuous stream of solution from the mixer 5 passes through a heat exchanger 6 to a pipe 7 leading to an insulated chamber 8, said pipe 7 being equipped with a pressure-reducing valve 9 to provide for a reduction in the pressure of the stream entering the chamber 8. The valve 9 is adjusted to provide for evaporation of portions of the normally gaseous solvent, thereby chilling the remaining oil solution to precipitate wax therein.

Any suitable normally gaseous dewaxing solvents may be employed in this system. Examples appear in propane, methane, ethane, and mixtures including these relatively light hydrocarbons. However, for convenience in describing the invention, we will refer to propane as a suitable normally gaseous solvent, without limiting the invention to this specific solvent.

To provide the desired ratio of solvent in oil to be dewaxed, a regulated stream of cold liquid propane may be transmitted through a pipe 10 to the pipe 7, said pipe 10 being equipped with a pump 11 and regulating valve 12. An advantage can be gained by introducing the propane from pipe 10 in a very cold condition, for example, at a temperature lower than about -40°F. , so as to suddenly chill the relatively warm solution to a temperature of about 10°F. More specifically stated, the incoming solution may pass from the heat exchanger 6 at a temperature of about 60°F. , and the shock chilling due to the stream from pipe 10 may suddenly chill the oil solution to a

temperature of about 10°F. , thereby instantly precipitating high melting point waxes. This preliminary chilling also reduces the chilling requirements in the chamber 8. However, in commercial practice, the chamber 8 may be relatively large to provide the desired capacity, and a series of such chambers may be employed, instead of using a single chamber 8.

Instead of adhering to the generally accepted critical rate of very gradual chilling, we preferably regulate the valve 9 to very suddenly reduce the pressure in a disintegrating zone formed by the chamber 8. An instantaneous reduction of this pressure will result in sudden evaporation of portions of the solvent, so as to suddenly chill and precipitate wax fractions in a liquid solution of the oil and solvent. Furthermore, this violent flash chilling will suddenly expand the evaporating portions of the solvent to produce an explosive effect, thereby forcibly disintegrating the wax. The oil solution is thus liberated from the wax, and there is no opportunity for the usual formation of wax clusters which occlude or trap portions of the oil solution in the wax.

Of course, this violent disintegration may not produce readily filterable wax. However, one of the objects of the invention is to dewax the oil without resorting to trouble and expense of employing the conventional filter plants, and as a preliminary step, we preferably disintegrate the wax to liberate all of the oil solution for the dewaxing operation.

The disintegrating chamber 8 is provided with a gas outlet pipe 13 at the top, and a valve 14 in said pipe may be regulated to control the discharge of gas. Any suitable recovery system may be employed to liquefy this gas for reuse in the system.

We will now refer to a settling chamber, or dewaxing chamber, 15 surrounded by the upper portion of an insulated column 16. An annular space may be formed around said settling chamber 15 to provide an outer settling chamber 17 to be hereinafter described. The chamber 15 may be open at the bottom to form a wax outlet 19, and the top of said chamber 15 may be provided with a pipe 20 having a valve 21 to regulate the discharge of dewaxed solution.

A continuous stream of disintegrated wax and oil solution is discharged from the bottom of the chamber 8, through a pipe 22 leading into the dewaxing chamber 15 as shown in the drawing. This pipe 22 is equipped with a pump 23 and valve 24 to regulate the flow of wax and oil solution to the dewaxing chamber 15. The incoming stream of cooled solution and wax may be delivered to this chamber at approximately the temperature selected for precipitation of wax in the disintegrating chamber 8. For example, in using propane to dewax lubricating oils, this temperature may be about -40°F. , although higher and lower temperatures may be employed in separating various kinds of waxy materials.

We are now assuming that a relatively light solvent has been selected for the dewaxing operation in the chamber 15, and that the viscosity and interfacial tension are low enough to allow the wax particles to descend in the relatively light solution. In this event, the major portion of the light solution will rise to the top of the dewaxing chamber 15, where it is discharged in the form of a continuous stream through the pipe 20. This outgoing solution is free of wax, and it may be distilled in any suitable manner to remove the solvent from the dewaxed oil.

Attention is now directed to the separating zone, or dewaxing zone, established near the point where the incoming mixture of cooled solution and wax begins to separate in the chamber 15. The disintegrated wax particles move downwardly and carry with them portions of the oil solution, including oily films and relatively free portions of the solution between the descending wax particles. The major portion of the light oil solution freely rises to the top of the chamber 15, but a minor portion of this oil solution is carried downwardly with the wax, which is discharged from a lower portion of said chamber 15.

Therefore, as a supplement to the dewaxing operation herein claimed, advantages can be gained by subjecting this outgoing wax to a simple deoiling operation. For example, the wax may be transmitted through a counterflowing stream of selective oil solvent to dissolve and recover oil solution carried by the wax.

To illustrate a suitable condition of this kind, we have shown a counterflow deoiling system including the insulated column 16 associated with the dewaxing chamber 15.

The mass of wax descending from the chamber 15 passes through a relatively light rising stream which may be produced by continuously introducing a stream of deoiling solvent through a pipe 25 leading into the lower portion of the column 16. The pipe 25 is equipped with a pump 26 and a regulating valve 27 adjusted to regulate the flow of the incoming deoiling solvent. The stream of deoiling solvent from the pipe 25 will rise in direct contact with the descending stream of wax particles, so as to selectively dissolve the free portions of oil solution carried by the wax, and also forcibly scrub the oily films from the descending wax particles. This action may be aided by any suitable mechanical appliances. For example, the column 16 may be provided with alternating baffles including a series of rings 28 extending inwardly from the inner face of the column 16 and a series of central baffle members 29 arranged as shown in the drawing to produce an elongated tortuous passageway wherein the descending wax particles move back and forth in a zigzag course. The stream of selective deoiling solvent rises through the descending stream of wax particles, and the counterflowing streams are divided into numerous layers of different specific gravities contacting with each other and advancing in intersecting paths as they flow from one baffle to another. These baffles are preferably provided with downwardly inclined top and bottom faces to permit free movement of the descending wax particles. It will be observed that the descending layers of wax particles move downwardly while in contact with the downwardly inclined top faces of the baffles, and that said wax particles repeatedly intersect the rising solvent while descending from one inclined plane to another. A continuous stream of deoiled wax with a minor portion of the solvent may be discharged through a pipe 30 below the inlet for the deoiling solvent.

The inclined faces of the baffles tend to prevent the wax particles from clinging to said baffles. However, any suitable mechanical means may be employed to prevent or limit the growing of wax crystals which might otherwise extend from the top faces of the baffles. For example, the central baffles 29 may be fixed to a slowly rotating shaft 31 so as to rotate with the shaft, and scrapers 32 may extend from bottom faces of these rotary baffles to remove waxy material from the tops of

the stationary baffle rings 28. These stationary baffles may be likewise provided with scrapers 33 extending from their bottom faces to remove waxy material from the top faces of the rotating baffles.

An interesting condition appears in the effective cleansing of the descending wax particles due to the forcible scrubbing and dissolving action of the selective deoiling solvent which repeatedly crosses the path of the wax in the deoiling zone. Attention is also directed to the condition at the upper portion of this deoiling zone where the rising solvent stream is diverted from the interior of the dewaxing chamber 15. This deflection of the rising solvent is due partly to the downward force of the descending stream of wax particles issuing from the bottom of said chamber 15, and partly to a rising current in the surrounding chamber or passageway 17. The highest central baffle 29 also tends to deflect the rising solvent toward the chamber 17. Some of the wax particles may be swept into the settling chamber 17, but these wax particles are relatively heavy, so they will eventually fall in the lighter solvent and return to the mass of wax, instead of passing to the top of said settling chamber 17.

The selective deoiling solvent performs a very important function in selectively removing oil carried by the wax particles. However, most of the oil is removed from the wax in the dewaxing chamber 15, so the deoiling solvent stream which rises through the settling compartment 17 contains a relatively small percentage of the original oil. Therefore, this reasonably fresh body of selective oil solvent can be very conveniently used in the dewaxing stage of the process. For example, it may be continuously transmitted from the top of the settling compartment 17 through a pipe 34 and through a pump 35 and valve 36 in said pipe 34 which leads to the heat exchanger 6, and thence through pipe 4 to the continuous incoming stream of wax-bearing oil in the pipe 1.

The velocity and time of the deoiling action may be regulated to dissolve practically all of the oil carried by the wax, or any desired percentage of oil may be discharged with the wax, depending upon the commercial requirements.

It is to be understood that the invention is not limited to the specific arrangements of chambers and zones herein disclosed to illustrate one form of the invention, and that any suitable solvents may be employed to produce the counterflow of wax and solvent in the deoiling system. However, when the deoiling solvent admitted through the pipe 25 is eventually transmitted to the incoming charge in the pipe 1, so as to serve as a dewaxing solvent, an advantage may be gained by selecting a solvent suitable for both operations. For example, if the primary dewaxing solvent is propane, a stream of fresh cold propane in a liquid state may be admitted through the pipe 25, and after performing its deoiling function in the counterflow system, this liquid propane and the oil dissolved therein, may be transmitted through the pipes 34 and 4 to the incoming stream of charging stock in the pipe 1. The oil recovered in the deoiling operation is thus very economically recycled to the charging stock, while the used deoiling solvent is employed as a solvent for the dewaxing operation.

This procedure enables a single solvent to very effectively perform two successive functions, while recycling recovered oil to the incoming charge. It also eliminates a distilling operation which would otherwise be required to separate the de-

oiling solvent from its recovered oil, and if any of the valuable wax should be incidentally dissolved in the deoiling solvent, such wax will be recycled to the incoming charging stock.

The term "disintegrating" as used in the claims refers to the effect of sudden expansion which forcibly separates a crowded mass of wax particles. In some cases, portions of the solvent may be securely trapped, or occluded, in relatively large clusters of the wax particles, so as to provide an internal bursting force when the pressure is suddenly reduced. In other cases, the "disintegrating" effect occurs in a crowded mass of individual wax crystals before they have an opportunity to unite in the form of relatively large wax clusters. For example, an intense flash chilling at the chamber 8 will produce a crowded mass of elementary wax crystals, but the "disintegrating" effect of the sudden expansion will forcibly disintegrate or scatter this crowded mass, so as to avoid the usual trapping of oil solution in the precipitated wax.

We claim:

1. In the art of using normally gaseous selective dewaxing solvents to chill oil solutions to dewaxing temperatures, the process of dewaxing oil and deoiling the wax which comprises mixing a wax-containing oil with liquefied normally gaseous dewaxing solvent under pressure, to form a dewaxing solution, reducing the pressure to evaporate portions of the solvent, thereby chilling the remaining oil solution to precipitate wax therein, transmitting a mixture of the wax and oil solution into a settling chamber, discharging a stream of dewaxed oil solution from an upper portion of said settling chamber, discharging a stream of wax with portions of the oil solution from a lower portion of said settling chamber to a counterflow deoiling system, transmitting a rising stream of liquefied normally gaseous solvent through a descending stream of wax in said deoiling system, deflecting the counterflowing streams of wax and solvent into intersecting paths in said counterflow deoiling system, so as to forcibly scrub oil solution from the wax, discharging the deoiled wax from a lower portion of said deoiling system, discharging a stream of the used solvent with its dissolved oil solution from an upper portion of said deoiling system while diverting the last mentioned stream from the contents of said settling chamber, and transmitting said last mentioned stream of liquefied normally gaseous solvent and dissolved oil solution to said wax-containing oil, so as to recycle the dissolved oil from said deoiling system and at the same time utilize the last mentioned solvent in forming said dewaxing solution.

2. In the art of using normally gaseous selective dewaxing solvents to chill oil solutions to dewaxing temperatures, the process of dewaxing oil and deoiling the wax which comprises mixing a wax-containing oil with liquefied normally gaseous dewaxing solvent under pressure to form a dewaxing solution, reducing the pressure to evaporate portions of the solvent, thereby chilling the remaining oil solution to precipitate wax therein, transmitting a mixture of the wax and oil solution into a settling chamber, discharging a stream of dewaxed oil solution from an upper portion of said settling chamber, discharging a stream of wax with portions of the oil solution

from a lower portion of said settling chamber to a counterflow deoiling system, transmitting a rising stream of liquefied normally gaseous solvent through a descending stream of wax in said deoiling system, dividing the rising and descending streams into numerous layers of different specific gravities contacting with each other at different elevations in said deoiling system, causing the counterflowing streams of wax and solvent to flow in intersecting paths while moving from one of said elevations to another, so as to forcibly scrub oil solution from the wax, discharging deoiled wax from a lower portion of said deoiling system, discharging a stream of the used solvent with its dissolved oil solution from an upper portion of said deoiling system while diverting the last mentioned stream from the contents of said settling chamber, and transmitting said last mentioned stream of liquefied normally gaseous solvent and dissolved oil solution to said wax-containing oil, so as to recover said oil solution and at the same time utilize the last mentioned solvent in forming said dewaxing solution.

3. In the art of using normally gaseous selective dewaxing solvents to chill oil solutions to dewaxing temperatures, a process of forcibly disintegrating the wax and thereafter deoiling the disintegrated wax, said process of disintegrating and deoiling including the steps of mixing a wax-containing oil with a liquefied normally gaseous dewaxing solvent under pressure to form a dewaxing solution, suddenly reducing the pressure in a disintegrating zone to produce forcibly disintegrated wax particles, thereby suddenly evaporating portions of the solvent to quickly chill and precipitate wax fractions in a liquid solution of the oil and solvent, while suddenly expanding the evaporating portions of the solvent in the waxy mixture to forcibly disintegrate the wax, transmitting a mixture of the disintegrated wax and oil solution into a settling chamber, discharging a stream of dewaxed oil solution from an upper portion of said settling chamber, discharging a stream of wax with portions of the oil solution from a lower portion of said settling chamber to a counterflow deoiling system, transmitting a rising stream of liquefied normally gaseous solvent through a descending stream of wax in said deoiling system, dividing the rising and descending streams into layers of different specific gravities contacting with each other at different elevations in said deoiling system, causing the counterflowing streams of wax and solvent to flow in intersecting paths while moving from one of said elevations to another, so as to forcibly scrub oil solution from the wax, discharging deoiled wax from a lower portion of said deoiling system, discharging a stream of the used solvent with its dissolved oil solution from an upper portion of said deoiling system while diverting the last mentioned stream from the contents of said settling chamber, and transmitting said last mentioned stream of liquefied normally gaseous solvent and dissolved oil solution to said wax-containing oil, so as to recycle the dissolved oil from said deoiling system and at the same time utilize the last mentioned solvent in forming said dewaxing solution.

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