

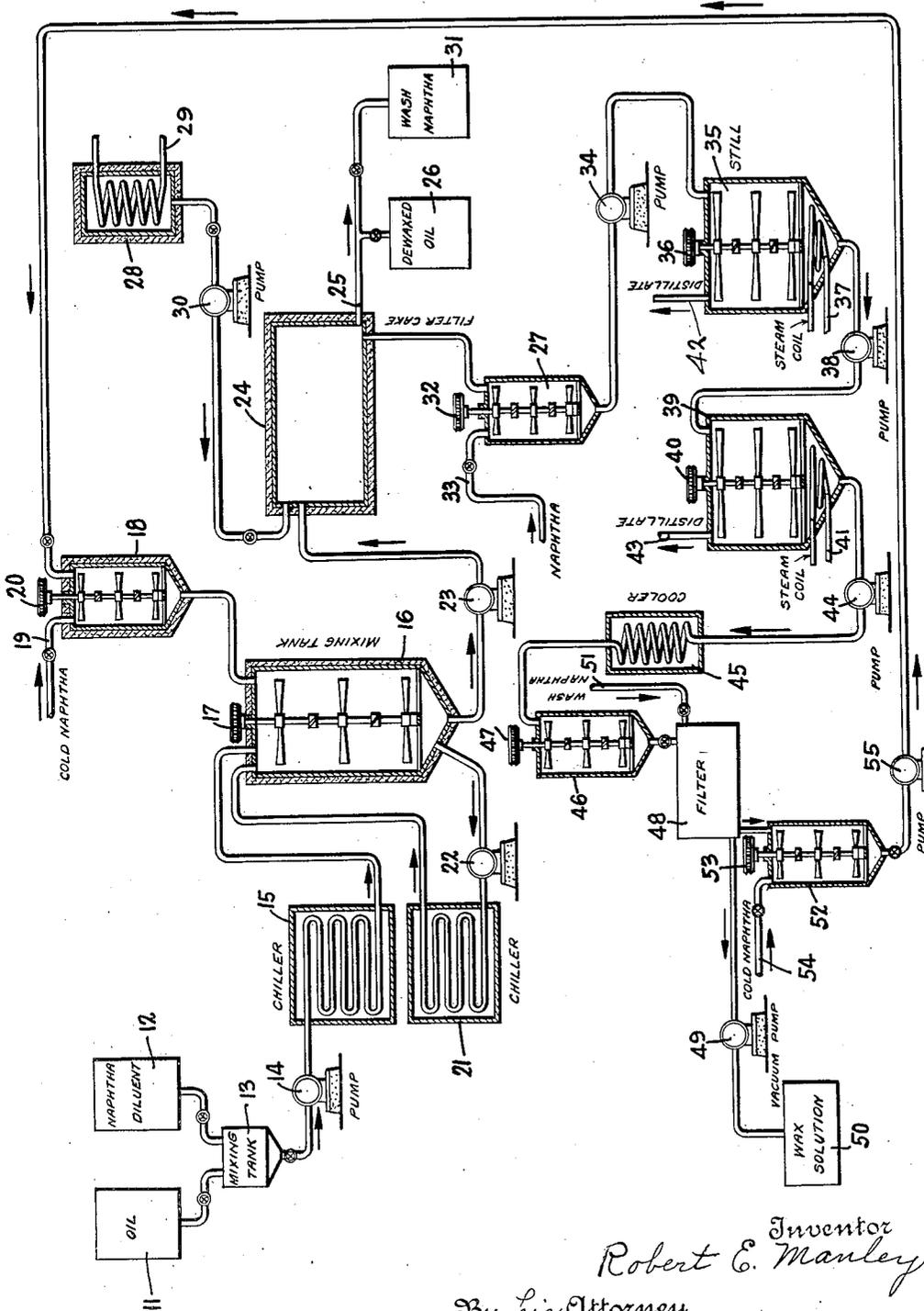
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TREATING HYDROCARBON OIL

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UNITED STATES PATENT OFFICE

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TREATING HYDROCARBON OIL

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This invention relates to a process for treating hydrocarbon oil and more particularly to a process of treating wax-bearing hydrocarbon lubricating oils to effect removal of solid and semi-solid waxy constituents whereby the temperature of congelation of the oil is reduced. Considered broadly the invention comprises a process wherein wax-bearing hydrocarbon lubricating oil, which may have been subjected to any desired preliminary refining treatment, is chilled to a temperature which will effect precipitation of the solid and semi-solid waxy constituents of the oil, after which the precipitated constituents are separated from the oil by filtration at temperatures below their precipitation temperature. To aid in the separation of waxy constituents from the oil, a comminuted solid material or "filter-aid" is mixed with the oil, the filter-aid and the precipitated wax being deposited together upon the filter. The wax and the filter-aid material are then separated and the filter-aid is dehydrated and used again in dewaxing further wax-bearing hydrocarbon oil.

The invention may be readily understood from a description of the procedure of the process considered in connection with the accompanying drawing which represents a diagrammatic sectional elevation of an apparatus which may be employed for carrying on the various steps of the process. The apparatus illustrated in the drawing is shown and described merely for the purpose of aiding in understanding the process and it is to be understood that any other suitable apparatus may be substituted for that shown.

Referring to the drawing, there is illustrated a source of oil to be dewaxed such as tank 11 and a source of a suitable diluent, for example naphtha, such as the tank 12. A mixing vessel 13 is provided wherein the wax-bearing oil and diluent may be mixed if so desired. From the mixing vessel 13 the wax-bearing oil, which may or may not be diluted, is forced by means of the pump 14 through the chiller 15. The chiller may be of any suitable design and is preferably the tubular type in which the oil passes through a pipe surrounded by a jacket through which cold

brine flows in a direction counter to the flow of oil. After having been chilled to the proper temperature to effect precipitation of the waxy constituents, the oil is introduced into the insulated mixing tank 16 fitted with a suitable mechanical agitating device 17 wherein the oil is thoroughly mixed with the proper quantity of a comminuted solid material or "filter-aid", such for example as diatomaceous earth. From the container 18 the filter-aid material may advantageously be supplied to the mixing tank in the form of a suspension or slurry by mixing with either naphtha or a portion of oil which has previously been dewaxed. Accordingly, a means for supplying cold naphtha to the tank 18 is provided in the line 19 leading from a suitable source (not shown). The tank 18 is fitted with mechanical agitating means provided to maintain the filter-aid in a substantially uniform suspension in the naphtha. Although the mixing tank 16 is heavily insulated there would normally be some loss in refrigeration to the atmosphere and accordingly an additional chiller 21 is provided and a pump 22 connected thereto through the lines as shown, whereby the contents of the tank 16 may be circulated through the chiller to maintain the desired low temperature in the tank 16.

From the mixing tank 16 the mixture of chilled oil and diatomaceous earth is forced by the pump 23 through the filter press 24 which is heavily insulated and which may be also brine-jacketed in order to maintain the chilled oil at the desired low temperature. The filter press may be of any suitable type and it has been found from experience that the type of pressure filter known as the Kelly filter is particularly satisfactory for this work. After passing through the filter the oil is discharged through the connection at 25 into the storage tank 26 while the precipitated wax and diatomaceous earth are deposited in a cake upon the filtering surfaces. A container 27 is supplied to receive the cake of wax and diatomaceous earth which may be periodically dumped from the filter. A source of supply for cold naphtha is provided, as the insulated tank 28, equipped

with a means for chilling such as the cooling coil 29, and a pump 30 is provided for forcing the cold naphtha through the filter for washing oil from the filter cake. A tank 31 with suitable connections is provided for receiving the naphtha after it has been used in washing the filter cake.

The tank 27 is fitted with an agitating device 32 and a connection 33 whereby naphtha may be supplied from a suitable source (not shown). When the filter cake is dumped into the tank 27 it is mixed and agitated with naphtha to form a suspension which is then pumped by means of the pump 34 through the connections as shown, to a still 35, fitted with agitating means 36. Heat is applied to the still by any suitable means, as for example the steam coil 37, the temperature being so elevated as to distill off a portion of the naphtha, which carries with it water absorbed by the diatomaceous earth during the dewaxing process. From the still 35 the partially dehydrated suspension of the diatomaceous earth in the naphtha solution of wax is pumped by means of the pump 38 into a second still 39, also fitted with agitating means 40 and heated by suitable means such as the steam coil 41, wherein distillation continues until the diatomaceous earth has been substantially dehydrated.

From the stills 35 and 39 the distilled products, consisting of naphtha and water, pass off to a suitable condensing system (not shown) through the connections 42 and 43, respectively. After condensation, the naphtha and water may be separated by a proper system of traps and the naphtha returned to the stills if so desired.

From the still 39 the dehydrated suspension is pumped by means of the pump 44 through a suitable cooler 45 to the tank 46 which is fitted with agitating means 47 to maintain the diatomaceous earth in suspension. The tank 46 serves as a supply tank for a filter 48 which may be of any suitable design such, for example, as that known as the American continuous filter. Herein the diatomaceous earth is deposited upon the filtering surfaces in the form of a cake while the solution of wax in naphtha is withdrawn from the filter by means of the vacuum pump 49 to a tank 50 where it may be stored for further disposition. The filter cake and diatomaceous earth may be washed by means of warm naphtha supplied through the connection at 51 from a suitable source (not shown).

The filter cake, which consists of diatomaceous earth, moisture-free, and in a form suitable for reuse in the dewaxing process, is discharged from the filter into the tank 52 which is fitted with agitating means 53. It may there be mixed with dewaxed oil or naphtha, preferably the latter, which should be cold and which may be supplied through

the connection 54 from a suitable source (not shown). The diatomaceous earth may then be pumped, in the form of a suspension, by means of pump 55 to the tank 18 from which it may be introduced at the proper time to the mixing tank 16 and re-used in the dewaxing of further oil.

The operation of the process in connection with the apparatus shown may be substantially as follows:

The oil to be dewaxed, for example a treated and filtered cylinder stock having a pour test of about 70° F., is introduced through the tank 11 into the mixing tank 13 where it is thoroughly mixed with a suitable diluent, preferably petroleum naphtha in proportion of about 30 to 60 per cent by volume of the diluent. The diluted oil is then forced through the chiller 15 by means of the pump 14 and the temperature is thus reduced to the degree desired to effect precipitation of the solid and semi-solid waxy constituents contained therein. The temperature to which the diluted stock is chilled will depend somewhat upon the degree of dewaxing which it is desired to obtain, which is in turn dependent upon the maximum temperature of congelation desired in the finished oil and, in general, it may be said that the oil should be chilled to a temperature several degrees lower than the congelation temperature desired in finished dewaxed oil. In the present instance it may be desired that the dewaxed cylinder stock have a pour test or congelation temperature not exceeding approximately 30° F., in which case it will be desirable to chill the diluted stock to about 15° to 25° F.

From the chiller 15 the diluted stock passes into the mixing tank 16 where it is mixed with a suitable filter-aid material such as diatomaceous earth, in the desired proportion, which is in this instance about 10 to 30 pounds of earth for each barrel of diluted stock. The diatomaceous earth is supplied from the tank 18, preferably in the form of a suspension in cold naphtha. It is of extreme importance that the diatomaceous earth supplied to the mixing tank 16 shall be substantially anhydrous. If the dewaxing process is carried on at temperatures below 32° F. any moisture present in the earth forms ice which fills up the porous structure of the earth rendering it so inefficient as to be practically worthless as a filter-aid in the process. In general, it will be found that a moisture content exceeding 5 per cent by weight will so greatly reduce the efficiency of the diatomaceous earth as a filter-aid as to materially impair the practical operation of the process when a recovered filter-aid of such high moisture content is used, while for the best results, the moisture content should not exceed 1 per cent by weight.

The earth is very finely powdered and the

contents of the tank 16 are continuously agitated by suitable mechanical means 17 to prevent settling of the precipitated wax and the earth. From the tank 16 the chilled diluted oil containing precipitated waxy constituents and diatomaceous earth in suspension is forced through the filter 24 by means of the pump 23. The dewaxed oil is discharged from the filter through suitable connections at 25 and passes to the storage tank 26, after which it may be treated in any suitable manner for the removal of the diluent to yield the finished dewaxed product.

The precipitated wax and diatomaceous earth are retained upon the filtering surfaces in the form of a more or less porous cake. When the proper thickness of cake has been built up on the filter the supply of oil from the tank 16 is cut off and the filter drained. The cake may then be blown with air and washed with cool naphtha from the tank 28 for the purpose of removing the last traces of oil, after which it may be dumped into the tank 27 where it is mixed with naphtha supplied through the connection at 33, the material being properly agitated by means of the agitator 32.

During the dewaxing operation described in the previous paragraph a certain amount of moisture is ordinarily absorbed from the oil by the diatomaceous earth. Further moisture condenses from the atmosphere and collects upon the cold filter cake during the time it is being discharged from the filter. As a result, the earth forming a part of the mixture in the tank 27, although originally introduced into the system in a dry state, may be found to have absorbed moisture up to as much as 75 per cent by weight or more. If such diatomaceous earth were merely separated from the wax portion of the filter cake and returned to the tank 18 for further use in dewaxing additional oil, it would be found to be practically worthless as a filter-aid material and the process would be substantially inoperative. Accordingly, the earth is recovered by a method involving dehydration which is as follows:

The contents of tank 27, consisting of a mixture of naphtha, wax, and diatomaceous earth, are forced by means of the pump 34 to the still 35. Heat is applied to the still through the steam coil 37, although any other suitable means for heating the still may be used, and the contents are maintained in suspension through the action of the agitating device 36. Distillation takes place in the still 35 and a portion of the naphtha and the water absorbed by the diatomaceous earth distills off through the connection 42. The still 35 may be so operated as to distill off all of the water contained in the diatomaceous earth but in the particular apparatus and example being described, an additional still 39 is provided and the contents of still 35

are accordingly heated to such an extent as to effect only partial dehydration of its contents, which are then transferred by means of the pump 38 to the second still 39 where distillation is again effected until the contents are thoroughly dehydrated.

It may be desirable to collect the distillates from the stills 35 and 39, separating the water from the naphtha by suitable separating means (not shown) and returning the naphtha to the stills in order that, through its distillation, more water may be carried off.

From the still 39 the dehydrated suspension of diatomaceous earth in the naphtha solution of wax is conveyed, by means of the pump 44, through the cooler 45 wherein it may be cooled to prevent substantial evaporation of the naphtha, after which it passes into the tank 46. Here the mixture is maintained in suspension by the agitating device 47 and is fed into the filter 48 where the dehydrated diatomaceous earth is deposited as a cake upon the filtering surfaces and may be washed free from wax by warm naphtha supplied through the connection at 51. The wax solution passing through the filter is collected in the tank 50, the naphtha contained therein being recoverable through distillation.

The filter cake, consisting of dewaxed and dehydrated diatomaceous earth, is discharged to the tank 52 where it may be mixed with naphtha, preferably cold naphtha, wherein it is maintained in suspension through the action of the agitating means 53. The diatomaceous earth in this condition is suitable for further use in dewaxing oil and, as desired, may be pumped by the pump 55 back to the tank 18 from which it may be introduced at proper intervals to the mixing tank 16 and there employed in dewaxing further hydrocarbon oil.

The specific example of the invention described in the preceding paragraphs has included a method of effecting dehydration of the recovered filter-aid material which consists in subjecting the material to distillation in the presence of a volatile fraction of petroleum, such as naphtha. However, other volatile solvents of wax may be used to replace the petroleum naphtha, for example, coal tar products and their derivatives, and also alcohols. It will be understood that the invention is not limited to a procedure involving dehydration by distillation with volatile solvents and that other suitable means for the removal of water from the filter-aid material may be used. For example, after the separation of wax from the filter cake obtained from the filter 24, the wax-free diatomaceous earth may be heated to temperatures sufficient to drive off the moisture. An equivalent procedure which may, under

certain circumstances, prove desirable, is to blow heated gases through the wax-free filter-aid material.

Obviously many modifications and variations of the invention, as hereinbefore set forth, may be made without departing from the spirit and scope thereof, and therefore, only such limitations should be imposed as are indicated in the appended claims.

10 I claim:

1. The process of dewaxing lubricating oil which comprises subjecting the oil to a separating treatment, while chilled and maintained at a low temperature whereby precipitation of wax constituents is effected and while intimately mixed with an inert comminuted solid filter-aid material containing not more than 5% by weight of free water comprising a filter-aid recovered from a previous operation of dewaxing lubricating oil, whereby said precipitated wax constituents and filter-aid material are separated from the oil, and finally separating the filter-aid material from the wax whereby the filter-aid may be re-used in a similar cycle of operations.

2. The process of dewaxing lubricating oil which comprises subjecting the oil to a separating treatment, while chilled and maintained at a low temperature whereby precipitation of wax constituents is effected and while intimately mixed with an inert comminuted solid filter-aid material containing not more than 1% by weight of free water comprising a filter-aid recovered from a previous operation of dewaxing lubricating oil, whereby said precipitated wax constituents and filter-aid material are separated from the oil, and finally separating the filter-aid material from the wax whereby the filter-aid may be re-used in a similar cycle of operations.

In witness whereof I have hereunto set my hand this 28th day of November, 1927.

ROBERT E. MANLEY.

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