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SEPARATION OF WAX FROM MINERAL OIL

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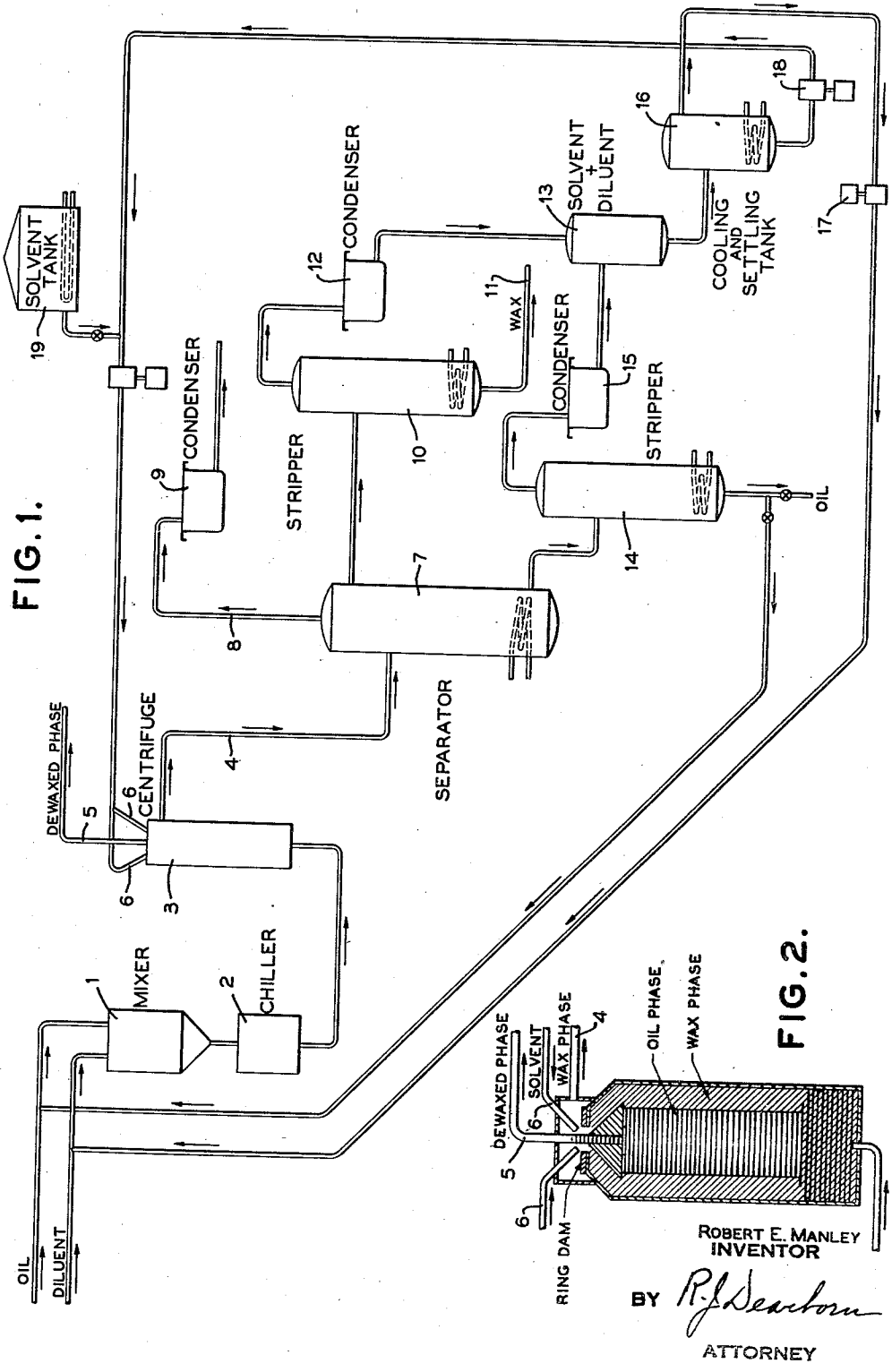


FIG. 1.

FIG. 2.

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SEPARATION OF WAX FROM MINERAL OIL

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4 Claims. (Cl. 196—18)

This invention relates to a method of separating wax from wax-bearing mineral oil.

It contemplates separation of wax from wax-bearing mineral oil by a method involving centrifuging wherein the wax is separated from the oil in the presence of a relatively high-boiling organic liquid. More particularly, the invention comprises a method of centrifuge dewaxing in which a relatively high-boiling solvent is introduced to the centrifuge bowl at a point adjacent the wax discharge therefrom. The solvent liquid acts as a carrier for the wax being separated from the oil, and also exerts a desirable solvent action upon the oil constituents associated with the wax as it is removed from the centrifuge. The solvent action is such that it facilitates the subsequent separation of this oil from the wax.

I contemplate using as the solvent liquid a selective solvent of the type such as furfural, phenol, tetrahydrofurfuryl alcohol, etc., or other relatively high-boiling solvents having a substantially high degree of solvent action upon the liquid hydrocarbon constituents of wax-bearing lubricating oil. The boiling point of the solvents mentioned above ranges from about 321° F. for furfural to 361° F. for phenol. I contemplate introducing the solvent to the wax phase discharge of the centrifuge bowl at an elevated temperature as, for example, around 300° to 320° F. However, it may be desirable to use the solvent at relatively lower temperatures, and even at temperatures approaching substantially the temperature of the wax-bearing oil within the centrifuge bowl.

I am aware that it has been customary heretofore in centrifuge dewaxing operations to employ a carrier liquid such as water or brine. In these prior art operations, it has been the practice, in some cases, to introduce the cold brine into the interior of the centrifuge bowl along with the charge. In other cases, it has been the practice to introduce hot water through the wax discharge port of the centrifuge bowl, or to apply a jet of hot water to the neck of the centrifuge bowl at a point adjacent the so-called ring dam.

The purpose of introducing either brine or water to the wax discharge port of the centrifuge bowl is to act as a carrier for the wax and facilitate its removal from the bowl of the centrifuge.

My invention contemplates using, in place of water, a relatively high-boiling organic liquid, such as furfural, which is, to a large extent, miscible with the oil contained in the wax phase leaving the centrifuge, thereby imparting fluidity to the mass and assisting in its discharge from the bowl. The solvent may be introduced to the centrifuge bowl at temperatures substantially higher than those used heretofore. Furthermore, the solvent has the additional advantage of aiding the subsequent separation of oil from the wax phase after removal from the centrifuge.

In the centrifuge dewaxing of wax-bearing mineral oil as heretofore practiced, the wax phase separated from the centrifuge comprises a petrolatum which contains, in addition to the wax, a substantial amount of oil. The object of my invention is to provide a means for recovering these valuable oil constituents contained in the petrolatum. I have, therefore, devised a method of utilizing as a carrier liquid one which also may be used to facilitate separation and recovery of the oil from the petrolatum in a subsequent operation which will be more fully described with reference to the accompanying flow diagram.

As shown in Figure 1 of the drawing, wax-bearing oil is conducted from a source not shown and introduced to a mixer 1. A diluent liquid such as naphtha is also conducted from a source not shown to this mixer. The mixture of oil and diluent is conducted through a chiller 2 wherein the mixture is chilled to any desired temperature sufficient to precipitate the wax constituents.

From the chiller 2, the chilled mixture is introduced to the centrifuge 3 which will be more fully described in connection with Figure 2.

The centrifuge bowl effects separation of the chilled mixture into a dewaxed oil phase and a wax phase. The wax phase, being of heavier specific gravity, is forced against the interior wall of the centrifuge bowl and removed through a discharge port 4 leading from the upper peripheral edge of the bowl. The dewaxed oil phase is discharged from the upper center of the centrifuge bowl through a port 5 and removed to separate tankage not shown. From such tankage it may be conducted to a suitable still for the removal of the diluent.

As is shown in more detail in Figure 2, the relatively high-boiling solvent liquid such as furfural is introduced to the centrifuge bowl through jets 6. This solvent is introduced preferably at a temperature of around 300° F.

The wax phase removed from the centrifuge bowl is conducted to a separator 7. The wax phase comprises a mixture of wax, solvent, diluent and oil. As already indicated, when the solvent is introduced to the centrifuge bowl at elevated temperature, the wax phase as removed therefrom will also be at a relatively high tem-

perature as, for example, about 270° F. The solvent will have dissolved in it substantially all or a large portion of the liquid hydrocarbon constituents thrown out with the wax in the centrifuge bowl.

Since the wax phase is thus at an elevated temperature, a portion of the diluent naphtha contained in it will flash into vapor upon introduction to the separator 7. Accordingly, the separator 7 is provided with a vapor outlet 8 and a condenser 9. The vaporized diluent is thus cooled and condensed in condenser 9 and removed for further use.

A coil placed within the lower portion of the separator 7 provides a means for controlling the temperature by passing through it a stream of hot or cold fluid depending upon the temperature required for the particular type of stock being treated.

The material remaining behind in the separator 7 will, upon standing, separate into two layers. The upper layer will comprise the wax mixed with a small amount of solvent and diluent liquids. The bottom layer will comprise the bulk of the solvent with oil dissolved in it and a small amount of diluent.

The upper layer is removed and conducted to a stropper 10 wherein the small amount of solvent and diluent remaining in the wax is stripped therefrom by heat in the presence of steam, or by the use of steam alone, leaving the wax in a substantially oil-free condition. This wax is removed from the stripper through a pipe 11, while the vaporized material is conducted through a condenser 12 and from there to a receiving tank 13.

The bottom layer accumulating in the separator 7 is conducted to a stripper 14 wherein the solvent liquid and diluent liquid is stripped away from the oil. The solvent and diluent vapors are removed through a condenser 15, and similarly conducted to the receiving tank 13. The oil collecting in the bottom of the stripper 14 may still contain a substantial amount of wax, and in that case it may advantageously be recycled through the system by returning it and mixing it with fresh wax-bearing oil entering the mixer 1.

The solvent and diluent liquid collecting in the tank 13 is withdrawn for the purpose of separating the diluent from the solvent. This may be accomplished by a distillation method. On the other hand, where the solvent is a compound such as furfural, and the diluent a compound such as naphtha, the separation may be effected by cooling to a temperature of around 30° to 80° F. At such temperatures, the two liquids are substantially immiscible and, therefore, separation will take place upon standing. In such case, the solvent and diluent mixture is withdrawn from the tank 13 to a cooling and settling tank 16, as shown, and the mixture cooled therein. The separated diluent liquid is withdrawn and returned by means of a pump 17 for mixing with fresh wax-bearing oil entering the mixer 1. The solvent liquid is likewise returned by a pump 18 to the solvent storage tank 19, and from which tank it is conducted in a heated condition to the bowl of centrifuge 3 by jet 6, as already described.

Referring now to Figure 2, which is merely a diagrammatic view of the interior of the bowl of centrifuge 3. As previously mentioned, the chilled wax-bearing mixture is introduced through the bottom of the bowl wherein, due to the effect of centrifugal action, separation into oil and wax phases occurs. The wax phase, being the heavier phase, is forced against the inner wall of the bowl, while the oil phase, and lighter phase, occupies the central portion of the bowl. The wax phase moves upwards against the inner wall of the bowl and discharges through the peripheral openings at the neck of the bowl into the outlet port 4. The oil phase discharges through the central port 5. The hot solvent is introduced through the jets 6 by means of which the solvent liquid is forced into the neck of the centrifuge bowl at the point at which the wax phase is discharging.

The hot liquid thus heats up the cold waxy material, rendering it substantially fluid and thus facilitating its rapid discharge from the centrifuge bowl.

Some of the hot liquid flows into the upper portion of the bowl and forms a liquid layer. Since it is heavier than the wax, it forms a film adhering to the wall of the bowl. This film, being at a relatively warm temperature, tends to melt the wax, converting it to a more fluid condition in the upper part of the bowl so that the movement of the wax in this portion of the bowl and through the discharge openings is expedited.

As a specific example, a lubricating oil fraction such as derived from Mid-Continent crude is mixed with naphtha in the proportion of about 30% oil to 70% naphtha. This mixture is chilled to a temperature of about —35° F. and introduced to the centrifuge 3 wherein it is subjected to centrifugal action at about that temperature.

Furfural heated to a temperature of 300° F. and in the proportion of about 1 part of furfural to 6.5 parts of cut-back wax-bearing solution charged to the centrifuge is introduced through the jets 6. On the basis of petrolatum separated it is desirable to use about 2 parts of furfural to 1 part of the separated petrolatum.

The dewaxed mixture of oil and diluent is removed through the central port 5 while the wax phase or petrolatum is withdrawn from the centrifuge through the port 4 together with hot furfural. The mixture, at a temperature of about 270° F. is introduced to the separator 7. A portion of the naphtha is flashed off as a vapor and removed through the condenser 9. The unvaporized material in the separator thus separates upon standing into two layers, as already explained, the upper layer comprising substantially all of the wax while the lower layer contains substantially all of the oil which was associated with the wax.

Due to variations in type of oil being dewaxed with respect to wax content, wax melting point, paraffinic nature or naphthenic character of the oil associated with the wax, it is desirable to use a carefully regulated temperature in separator 7. This is accomplished by the heat exchange coil installed in the separator 7.

The two layers are removed as already explained, and the solvent and diluent liquids associated therewith are removed by vaporization in separate operations, as fully described above.

While furfural has been mentioned, it is contemplated that other high-boiling organic solvent liquids of this general type may be used as, for example, phenol or tetrahydrofurfuryl alcohol. It is also contemplated that other diluent liquids may be used in place of naphtha as, for example, other petroleum fractions. Other types of dewaxing solvents may also be used as, for example, a mixture of acetone and benzol.

The acetone and benzol type of dewaxing sol-

vent is used to advantage where the oil to be dewaxed contains a large portion of wax which precipitates in distinctly crystalline form. This type of wax is difficult to remove in a centrifuging operation. Consequently, when a carrier liquid of the type mentioned above is employed, it offers an advantage in that the hot carrier liquid is miscible to a large extent with the wax phase. As a result, it increases the fluidity of the wax phase and thus facilitates its removal from the centrifuge.

There is a further advantage by employment of a carrier liquid such as furfural, and that is in the prevention of corrosion of the interior of the centrifuge bowl where the oil undergoing centrifuging has been subjected to a previous contacting treatment with acid-treated clay. Usually such oil retains small amounts of sulphur dioxide, and where this oil is centrifuged in the presence of water as a carrier liquid, a certain amount of corrosion naturally occurs. By employing furfural as the carrier liquid in place of water, this corrosion effect is thus eliminated.

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.

I claim:

1. The method of dewaxing wax-bearing mineral lubricating oil by continuous centrifugal separation wherein the wax is separated as a heavier phase and the oil as a lighter phase, comprising diluting the oil, chilling the dilute mixture to precipitate wax, passing the chilled mixture to a centrifuge, centrifugally separating the charge into an oil phase and a wax phase heavier than said oil phase within the bowl of the centrifuge, disposing a film of hot furfural over the interior wall of the centrifuge bowl thereby providing a carrier liquid for the separated wax phase, moving the separated wax phase by flotation upon said film of carrier liquid to the point of discharge from the bowl, separately withdrawing said phases from the bowl, and discharging the separated wax from the centrifuge bowl in the presence of a further quantity of hot furfural.

2. The method of dewaxing wax-bearing mineral lubricating oil by continuous centrifugal separation wherein the wax is separated as a heavier phase and the oil as a lighter phase, comprising mixing the oil with a dewaxing solvent, chilling the mixture to precipitate wax, passing the chilled mixture to a centrifuge, centrifugally separating the mixture into an oil phase and a wax phase heavier than said oil phase within the bowl of the centrifuge, introducing to the centrifuge at a point adjacent the point of wax phase discharge from said centrifuge bowl a carrier liquid comprising furfural at a temperature of around 270 to 300° F., disposing a film of hot furfural over the interior wall of the centrifuge bowl thereby providing a carrier liquid for the separated wax phase, moving the separated wax phase by flotation upon said film of carrier liquid to the point of discharge from the bowl, separately withdrawing said phases from the bowl, and discharging the separated wax phase from the centrifuge bowl in the presence of a further quantity of hot furfural.

3. The method of dewaxing wax-bearing mineral lubricating oil by continuous centrifugal separation wherein the wax is separated as a heavier phase and the oil as a lighter phase, comprising mixing the oil with a dewaxing solvent, chilling the mixture to precipitate wax, passing the chilled mixture to a centrifuge, centrifugally separating the mixture into an oil phase and a wax phase heavier than said oil phase within the bowl of the centrifuge, introducing to the centrifuge at a point adjacent the point of wax phase discharge from said centrifuge bowl a carrier liquid comprising furfural at a temperature of around 270 to 300° F., disposing a film of hot furfural over the interior wall of the centrifuge bowl thereby providing a carrier liquid for the separated wax phase, floating the separated wax phase on said film of carrier liquid to the point of discharge from the bowl, separately withdrawing said phases from the bowl, discharging the separated wax phase from the centrifuge bowl in the presence of a further quantity of hot furfural, subjecting the discharged wax phase to settling while at elevated temperature to separate into an upper layer comprising mainly wax with some furfural and dewaxing solvent and a lower layer comprising mainly oil, furfural and dewaxing solvent, removing said layers and recovering the furfural and dewaxing solvent therefrom.

4. The method of dewaxing wax-bearing lubricating oil by continuous centrifugal separation wherein the wax is separated as a heavier phase and the oil as a lighter phase, comprising mixing the oil with a dewaxing solvent, chilling the mixture to precipitate wax, passing the chilled mixture to a centrifuge, centrifugally separating the mixture into an oil phase and a wax phase heavier than said oil phase within the bowl of the centrifuge, introducing, at a temperature of around 270 to 300° F., to said centrifuge bowl an organic solvent liquid having a boiling temperature of the order of 321° F. and above and a specific gravity substantially greater than that of the wax phase, disposing a film of said organic solvent liquid over the interior wall of the centrifuge bowl thereby providing a carrier liquid for the separated wax phase, separately withdrawing said phases and discharging the separated wax phase from the centrifuge bowl in the presence of a further quantity of said hot organic solvent liquids.

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