METHOD OF PURIFYING WAX

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Fig. 1.

Fig. 2.

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METHOD OF PURIFYING WAX

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4 Claims. (Cl. 198—28)

This invention relates to a method of washing wax crystals to remove oil and other impurities therefrom. When wax bearing oils are dewaxed in accordance with any of the well-known dewaxing processes, the crude wax thus obtained contains a relatively large percentage of oil, generally 50% or higher. In order to produce a commercial grade of wax, it is necessary that a large percentage of this oil be removed from the wax.

Various methods for removing this oil have heretofore been practiced, the most common of which is the process known as sweating. All of the known processes for removing oil from wax are objectionable in that they require relatively large amounts of equipment and space, and the sweating process in particular requires a great deal of time, generally several days to a week.

It is therefore an object of this invention to provide an improved method of separating oil from wax which is simple in operation and requires simple and inexpensive equipment. Other and further objects will be apparent as this description progresses.

In the drawing, attached hereto and forming a part of this specification:

Fig. 1 is a diagrammatic representation of a form of apparatus adapted for use in practicing my process.

Fig. 2 is a diagrammatic representation of a modified form of apparatus for practicing my process.

The crude wax which may be obtained from any of the known dewaxing processes, and which contains approximately 50% of oil, is first mixed with a light liquefied normally gaseous hydrocarbon, preferably propane or some other liquefied normally gaseous hydrocarbon such as propylene, butane, iso-butane, butylene, ethane and ethylene, or mixture of the same, or other solvents known to have selective wax solvent properties at low temperature, whose density is not substantially greater than .6 at 0°F., and preferably is not substantially greater than that of propane. The amount of propane which is added to the crude wax is not more than an amount sufficient to make a slurry which may be readily handled. If the crude wax was obtained by a propane dewaxing operation in which the separation of wax from oil was accomplished by settling or centrifugation, so that the crude wax was obtained in the form of a slurry, this slurry itself may be used. The slurry of crude wax in propane is introduced through line 1 to the upper portion of tank 2 which is filled with liquefied propane at a temperature sufficiently low so that the waxes will be precipitated in the form of their crystals. The exact temperature of the bath of propane will of course vary with the nature of the waxes being treated and those which it is desired to recover. The temperature of the bath may be as low as -45°F. when it is desired to recover all the waxes and may be as high as -30°F. when only the high melting point wax is to be recovered.

Chilled propane is admitted through line 3 to 10 the lower portion of tank 2 and rises through tank 2 and goes out through line 4. The wax crystals introduced with this slurry through line 1 settle through the bath of cold propane in tank 2 and collect at the bottom, from whence they are discharged through line 5 in the form of a slurry. The bath of cold propane in tank 2 will dissolve most of the oil contained in the wax crystals, and will carry this oil out through line 4 in the form of a propane-oil solution. Due to the large body of liquefied propane maintained in tank 2, practically all of the oil will be dissolved or leached from the wax crystals, so that the wax crystals which fall to the bottom of tank 2 will be relatively pure and will then contain only minor proportions of oil. The pressure on the bath of cold propane in tank 2 is preferably maintained above the vapor pressure of the propane at the temperature in tank 2 by regulating valve 6 in line 4, so that there will be no bubbling of the liquid in tank 2 to hinder the settling of wax. The wax slurry discharged through line 5 may be subjected to filtration to remove most of the propane and any oil in solution with the propane, and then distilled to remove any remaining propane from the purified wax. As shown in the drawing, the slurry from line 5 is discharged on to a belt 18, which may be a filter cloth which passes between rollers 11 and 12 to squeeze out the oil and propane which falls into tank 13; the wax, which remains on belt 18, is discharged as the belt passes over roller 14 and falls into wax tank 15, as described in my copending applications, Serial No. 68,613 filed March 13, 1936.

As an alternative procedure the slurry obtained from line 5 may be introduced into a second tower similar to tower 2, the slurry in this case taking the place of the blend above described, which is introduced through line 1 into tower 2. In my copending application, Serial No. 68,610, filed March 13, 1936, I have described a process in which the crude wax, while in the filter media, is washed by solvent at gradually increasing temperature. However, it is possible to treat the wax in accordance with the present invention in a
manner similar to that described in my afore
mentioned application.
When it is desired to so treat the wax, the
temperature of the blend introduced into the
second tower may be increased slightly, either
by heat exchange or mixing additional warmer
propene with the slurry, and the temperature of
the propene forming the body of liquid con
tained in the second tower is increased slightly
over that of the propene in the first tower, pref
erably 5-20° F.

The apparatus shown in Fig. 2 is a dia
grammatic representation of a form of apparatus
adapted for carrying out this alternative pro
cedure. The chilled slurry of wax crystals in a
solvent is introduced into the upper portion of
a bath of chilled solvent in tower 16, through
valved line 18, while chilled solvent is introduced
into the lower portion of tower 16 through valved
line 20. The chilled solvent in tower 16 rises
slowly and dissolves any oil contained in the wax
slurry introduced through line 18. The solution
of solvent and oil is removed from tower 16
through valved line 21. The wax crystals fall
through the bath of chilled solvent and are re
moved from the bottom of tower 16 through
valved line 22 in the form of a wax slurry. The
temperature of the bath of chilled solvent in
tower 16 is preferably maintained sufficiently
low so that substantially all of the wax is pre
cipitated as wax crystals. The wax slurry re
moved through valved line 22 is introduced into
the upper portion of a bath of chilled solvent
maintained in tower 17, while additional solvent
is introduced to the lower portion of tower 17
through valved line 23. The temperature of the
solvent introduced through valved line 23 and of
the bath of solvent in tower 17 is somewhat high
er than that in tower 16, preferably 5-20° F.,
higher.

At this increased temperature the lower mel
ting point waxes will be taken into solution by
the chilled solvent and will be removed from
tower 17 through valved line 24. The higher
melting point waxes which are not dissolved by
the solvent fall to the bottom of tower 17 and are
removed through valved line 25 and introduced
into the upper portion of a bath of chilled solvent
maintained in tower 18. Additional solvent is
introduced to the lower portion of tower 18
through valved line 26. The temperature of the
solvent thus introduced and of the bath of the
solvent in tower 18 is somewhat higher than the
temperature maintained in tower 17. At this
temperature the medium melting point waxes
contained in the slurry introduced through valved
line 25 will be dissolved by a bath of chilled sol
vent and will be removed through valved line 27
in solution with the solvent. The higher melting
point waxes which are not dissolved by the sol
vent settle to the bottom of tower 18 and are
removed therefrom through valved line 28. The
solution of solvent with oil or wax dissolved therein, which are withdrawn from the top of
each tower, are collected separately and the sol
vent may be separated from the material dis
solved therein in any desired manner as by dis
tillation. There are thus obtained as many frac
tions of wax of different melting points as there
are settling towers. It is, of course, to be under
stood that any desired number of settling towers
may be connected in series.

I am aware that it has heretofore been pro
posed to wash crude wax crystals by permitting
them to settle through a bath of cold naphtha.
However, this prior process has not been adopted
since the difference in density between the wax
crystals and naphtha was not great enough to per
mit the rapid settling of wax which is necessary, if the
process is to be a continuous and economic one.
In fact, when it is attempted to use naphtha as
the solvent it is frequently almost impossible to
obtain any noticeable settling. Satisfactory op
eration of my process demands that the rate of
upward flow of fresh solvent must be great enough
to overcome the tenacity of the oil to diffuse
downwardly and that the rate of settling of the
wax crystals must be greater than the rate of
upward flow of the solvent, so that downward
setting will take place. These conditions are
not economically possible with naphtha but are
attainable with propene since the settling rate of
wax through propene is more than ten times as
fast as through naphtha.

A working equation (Stokes's) for finding the
velocity at which the wax crystals would settle
in a solvent is as follows:

\[
\frac{v}{\mu} = \frac{K(d_2 - d_1)^2}{d_3}
\]

where \(v\) = velocity of fall of particle.
\(K\) = a constant.
\(d_1\) = density of falling particle.
\(d_2\) = density of medium.
\(r\) = radius of falling particle.
\(\mu\) = viscosity of medium.

Assuming constancy of particle size, then for
60° A.P.I. naphtha at 0°F.

\[
V_s = \frac{0.9}{0.8} = 0.756
\]

For propane at 0°F.

\[
V_s = \frac{0.9}{0.12} = 5.83
\]

From the above calculations it is apparent that
by using chilled propene as the solvent both the
settling rate will be sufficiently high so that re
latively large quantities of crude wax may be con
tinuously purified by this method.

What I claim and desire to protect by Letters
Patent is as follows:

1. The method of fractionating a crude wax
into a plurality of waxes of varying melting points
which comprises forming a slurry of said crude wax
in a diluent having selective wax solvent
properties at low temperatures and having a dens
ity not substantially greater than 0.6 at 0°F.,
chilling said slurry to a temperature at which
substantially all of the wax will be precipitated in
the form of wax crystals, introducing said chilled
slurry into the upper portion of the first of a se
ries of settling zones, maintaining a body of
chilled diluent having the properties specified in
each of said settling zones, introducing said chil
ded diluent into the lower portion of each of
said settling zones, withdrawing a wax slurry
from the bottom of each of said settling zones,
introducing the slurry thus withdrawn into the
upper portion of the next succeeding settling
zone, maintaining the body of diluent in each of
said settling zones at a temperature higher than
that of the preceding zone to cause said diluent
in each successive settling zone to dissolve suc
cesive fractions of wax of increasingly higher
melting point from wax slurry introduced there-

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into, and withdrawing a solution of diluent with dissolved matter therein from the top of each of said settling zones.

2. The method of fractionating a crude wax into a plurality of waxes of varying melting points which comprises forming a slurry of said crude wax in a diluent having selective wax solvent properties at low temperatures and having a density not substantially greater than .6 at 0° F., chilling said slurry to a temperature at which substantially all of the wax will be precipitated in the form of wax crystals, introducing said chilled slurry into the upper portion of the first of a series of settling zones, maintaining a body of chilled diluent having the properties specified in each of said settling zones, introducing said chilled diluent into the lower portion of each of said settling zones, withdrawing a wax slurry from the bottom of each of said settling zones, introducing the slurry thus withdrawn into the upper portion of the next succeeding settling zone, maintaining the body of diluent in each of said settling zones at a temperature higher than that of the preceding zone to cause said diluent in each successive settling zone to dissolve successive fractions of wax of increasingly higher melting point from wax slurry introduced thereto, withdrawing a solution of diluent with dissolved matter therein from the top of each of said settling zones, and maintaining the pressure in each of said settling zones above the vapor pressure of the diluent at the temperature maintained therein.

3. The method of separating oil from wax and fractionating the wax into a plurality of waxes of varying melting points which comprises adding to the oil-wax mixture a diluent having selective wax solvent properties at low temperature and having a density not substantially greater than .8 at 0° F., chilling to obtain crystallization of the wax, introducing the chilled mixture into the upper portion of the first of a series of settling zones, maintaining in each of said settling zones a body of chilled diluent having the characteristics specified, introducing said chilled diluent into the lower portion of each of said settling zones, withdrawing a solution of diluent with dissolved matter therein from the top of each of said settling zones, withdrawing a solution of diluent with dissolved matter therein from the top of each of said settling zones, introducing the slurry thus withdrawn from each zone except the last into the upper portion of the next succeeding settling zone, maintaining the body of diluent in each zone following the first zone at a temperature higher than that of the preceding zone to cause said diluent in successive settling zones to dissolve successive fractions of wax of increasingly higher melting point, and withdrawing a solution of diluent with dissolved matter therein from the top of each of said settling zones.

4. The method of separating oil from wax and fractionating the wax into a plurality of waxes of varying melting points which comprises adding to the oil-wax mixture a diluent having selective wax solvent properties at low temperature and having a density not substantially greater than .6 at 0° F., chilling to obtain crystallization of the wax, introducing the chilled mixture into the upper portion of the first of a series of settling zones, maintaining the body of diluent in each zone following the first zone at a temperature higher than that of the preceding zone to cause said diluent in successive settling zones to dissolve successive fractions of wax of increasingly higher melting point, and withdrawing a solution of diluent with dissolved matter therein from the top of each of said settling zones, maintaining the pressure in each of said settling zones above the vapor pressure of the diluent at the temperature maintained therein.

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