

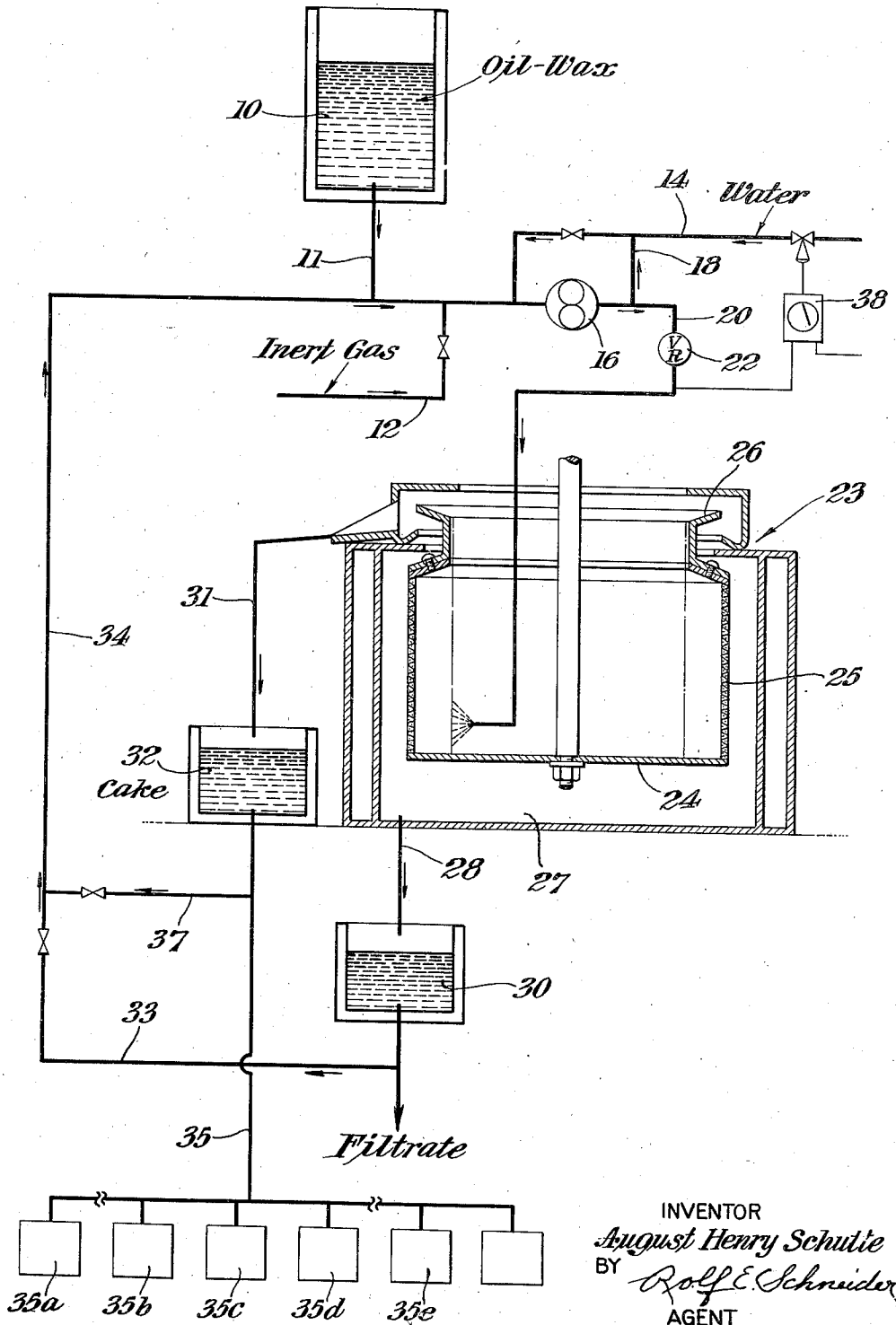
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METHOD OF SEPARATING WAX

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METHOD OF SEPARATING WAX

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This invention relates to improvements in the method of obtaining particular melting point waxes and similar normally solid crystalline masses from a charge of such materials with other materials of a different melting point, and more particularly relates to the separation by differences in freezing point rather than fractionation by differences in boiling points of various mixtures of materials.

As is pointed out in my prior patent, No. 2,168,142, I have found it entirely satisfactory to separate different melting point waxes from a slack wax or a crude scale wax by a new method which is characterized more particularly by the formation of an emulsion or intimate dispersion of a waxy mixture in nonsolvent, immiscible gaseous and liquid materials such as air and water. It appears that such emulsion or agitation tends to reduce the viscosity of the lower melting point materials such as the oils, and to form a fine dispersion of the higher melting point materials in an air environment which tends to give an unexpected release of the liquid portion from the solid portion. It is thus possible for the first time to obtain a commercial separation of materials of relatively close melting and freezing points and which are normally partially soluble in one another such as the hydrocarbon waxes.

It is necessary, however, in the commercial production of specification waxes, which are referred to herein as normally solid materials, by separation of the foots oil, which is normally liquid, to meet other requirements besides oil content, such as the tensile strength, and for this reason it may be necessary to blend waxes of different melting points and different oiliness content to obtain the specified material.

The principal object of my invention is to provide an improved method of separating different melting point materials by the formation of an intimate dispersion or emulsion thereof and to produce specified characteristics of the final product.

A further object of my invention is to provide an improved method of obtaining sharp fractionation of different melting point materials by differences of their freezing point and in which the differences in melting point may be as low as one or two degrees.

A still further object of my invention is to improve the control of the separation of different melting point waxes from waxy hydrocarbon fractions and mixtures thereof.

Further objects and advantages of my invention will appear from the following description

of a preferred form of embodiment thereof, taken in connection with the attached drawing illustrative thereof and in which the figure is a diagrammatic flow diagram of the apparatus by which my invention may be carried out.

In accordance with the preferred form of embodiment of my invention, a charge material of a mixture of different melting point materials such as oil and wax in the tank 10 is liquefied as by heating and the liquid mixture is withdrawn through the line 11. An inert gas such as air is fed into the mixture through line 12, and a non-solvent and normally immiscible liquid such as water is also introduced to the charge through the line 14. The mixture is then agitated in a suitable emulsifying, homogenizing or other mixing device 16.

The mixing device 16 is adapted to disperse the higher melting point material in the liquid materials, and by recycling a portion through line 18 and by the combined effect of the cooling accomplished by the water, a material of the character of shaving lather is discharged through line 20 against the pressure of relief valve 22.

The characteristic of this so-called dispersion or emulsion is rather unique in that it appears to be a plastic mass having a large number of air channels, the air of which appears to surround and perhaps to be adsorbed by the crystallized and precipitated higher melting point materials. These air channels serve as the passages for the liquid which is evidently an emulsion of water and oil. While I do not desire to be limited to any theory of operation, it is clear that the so-called aerogel of wax and emulsion of oil and water tends to form a mixture in which the oil is much freer to leave the wax, and the wax becomes a highly dispersed structure so that the emulsion or dispersion may be readily separated by suitable liquid-solid separating means.

More particularly, I prefer to separate this material in a form of centrifugal separator generally indicated at 23, which may be provided with a rotating basket 24 having a perforated side wall 25, through which the liquid materials are discharged by the centrifugal force. The cake formed is highly porous and light and may be washed with water or other displacing or solvent agents. I have found that the cake will continuously discharge over the upper lip 26 of the separator, which is apparently due to the displacement of the relatively light, porous cake by the relatively heavy and dense liquid-solid dispersion or emulsion. It will be apparent that the amount of oil removed from the cake will

reduce its density, and the cake does not tend to collapse but to move upward at a satisfactory rate.

The filtrate may be collected in the chamber 27 and drawn off through the line 28 into the filtrate tank 30, and the cake is conveniently discharged through line 31 to a cake tank 32.

The melting point of the cake is a function of the melting point of the mixture and a function of the operating temperatures. The nearer the charge temperature is to the higher melting point material, the less precipitation of the higher melting point materials and the less yield of higher melting point material there will be. The yield of higher melting point material will be nearer one melting point, however. I have now found it possible by continuously recycling all of the filtrate 30 through the lines 33 and 34 back to the charge line 11 to obtain a separation of cakes of different melting points which may be discharged through line 35 into the separate tanks 35a, 35b, 35c, 35d, 35e, etc. In this manner, all of the different melting point materials can be segregated with any desired degree of accuracy.

One manner of controlling the temperature is by the use of a temperature controller 38 interconnected in the cooling water line 14. Such controller may be set to give periodic temperature reductions on the emulsion charge to the separator 23. If the temperature is reduced, it will be apparent that there will be a greater amount of cake, but the cake will be of a greater range of melting points. By slowly reducing the temperature and discharging the different melting point cake into the different cake tanks 35a, 35b, etc., as great a number of melting point products as desired can be obtained.

The amount of material for each cut will, of course, depend to a certain extent on the amount of material of the different melting points in the original charge. It will be apparent, however, that by adjusting the temperature through controller 38 and thus continuously reducing the temperature of the charge, any desired number of fractions of closely controlling melting point range will be produced. The lower temperature filtrate at each reduction in feed temperature will also aid to reduce the temperature of the emulsion feed.

It is possible to obtain a specification product by the desired blending of the different melting point materials. The high degree of separation of oil accomplished by my process provides such dry wax as to make the further sweating difficult, if not impossible. It is, therefore, desirable to provide this blending of waxes which cannot be sweated to obtain the desired specification material.

In one case, for example, an average melting point cake of 135° F. and having a broad wax cut varying from 122° F. to 147° F. was introduced as feed and the temperature of the charge was adjusted to produce a wax of 136° F. Thereafter the temperature of the operation was continued until 16 different fractions were obtained varying from 131.9° F. to 137° F.

The recycle of filtrate also makes it possible to more carefully control the quality of the emulsion if, due to the amount of solids present, it would be too stiff. When the amount of solids comprises approximately 60% of the charge, for example, cavitation frequently occurs. This appears to be due to the fact that instead of discrete wax particles which can pass through the

emulsifying mechanism 16 as a slurry in the oil and the emulsifying agent, a system may be obtained in which the wax crystals are macerated and mashed back into the oil by mechanical action of the mixer. A control of the recycle will, of course, prevent the so-called cavitation in the pump and will materially increase its volumetric capacity. This thus permits the use of the simplified rotary type mixer.

Alternatively it may be desirable to recycle some of the cake as through the line 37 if the amount of solids in the feed is so small as to prevent the formation of the desired emulsion.

While I have shown and described a preferred form of embodiment of my invention, I am aware that modifications may be made thereto, and I, therefore, desire a broad interpretation of my invention within the scope and spirit of my invention and of the claims appended hereinafter.

I claim:

1. In a continuous wax separation process including the continuous forming of an emulsion of different melting point waxes, a chilling non-solvent liquid and an inert gas; the steps for obtaining different melting point wax fractions from said emulsion, comprising subjecting the emulsion progressively to a continuous liquid-solid separating operation to separate a solidified higher melting point one of said waxes from the liquid of the emulsion, continuously and repeatedly recycling through said separation step and said emulsifying step the liquid including lower melting point waxes in a liquid condition separated from said solidified wax, progressively introducing chilling non-solvent liquid directly to said recycled liquid in the course of recycling of the latter to reduce the temperature of the recycled liquid step-by-step and solidify the different melting point waxes in succession, successively separating, in said separating step, the so-solidified waxes from the remaining liquid undergoing said recycling, and segregating from each other the different melting point wax fractions so obtained.

2. In a continuous wax separation process including the continuous forming of an emulsion of different melting point waxes, a chilling non-solvent liquid and an inert gas; the steps for obtaining different melting point wax fractions from said emulsion, comprising subjecting the emulsion progressively to a continuous liquid-solid separating operation to separate a solidified higher melting point one of said waxes from the liquid of the emulsion, continuously and repeatedly recycling through said separation step and said emulsifying step the liquid including lower melting point waxes in a liquid condition separated from said solidified wax, progressively introducing to said emulsifying step additional quantities of said chilling non-solvent liquid and emulsifying the recycled liquid therewith, regulating said introduction of chilling liquid to reduce the temperature of the recycled liquid step-by-step and solidify the different melting point waxes in succession, successively separating, in said separating step, the so-solidified waxes from the remaining liquid undergoing said recycling, and segregating from each other the different melting point wax fractions so obtained.

3. In a continuous wax separation process including the continuous forming of an emulsion of different melting point waxes, a chilling non-solvent liquid and an inert gas; the steps for obtaining different melting point wax fractions from said emulsion, comprising subjecting the

emulsion progressively to a continuous liquid-solid separating operation to separate a solidified higher melting point one of said waxes from the liquid of the emulsion, continuously and repeatedly recycling through said separation step and said emulsifying step the liquid including lower melting point waxes in a liquid condition separated from the solidified wax, progressively introducing additional chilling non-solvent liquid to said recycled liquid in the course of recycling 10

the latter and emulsifying the recycled liquid with said added chilling liquid to reduce the temperature of the recycled liquid step-by-step and solidify the different melting point waxes in succession, successively separating, in said separation step, the so-solidified waxes from the remaining liquid undergoing said recycling, and segregating from each other the different melting point wax fractions so obtained.

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