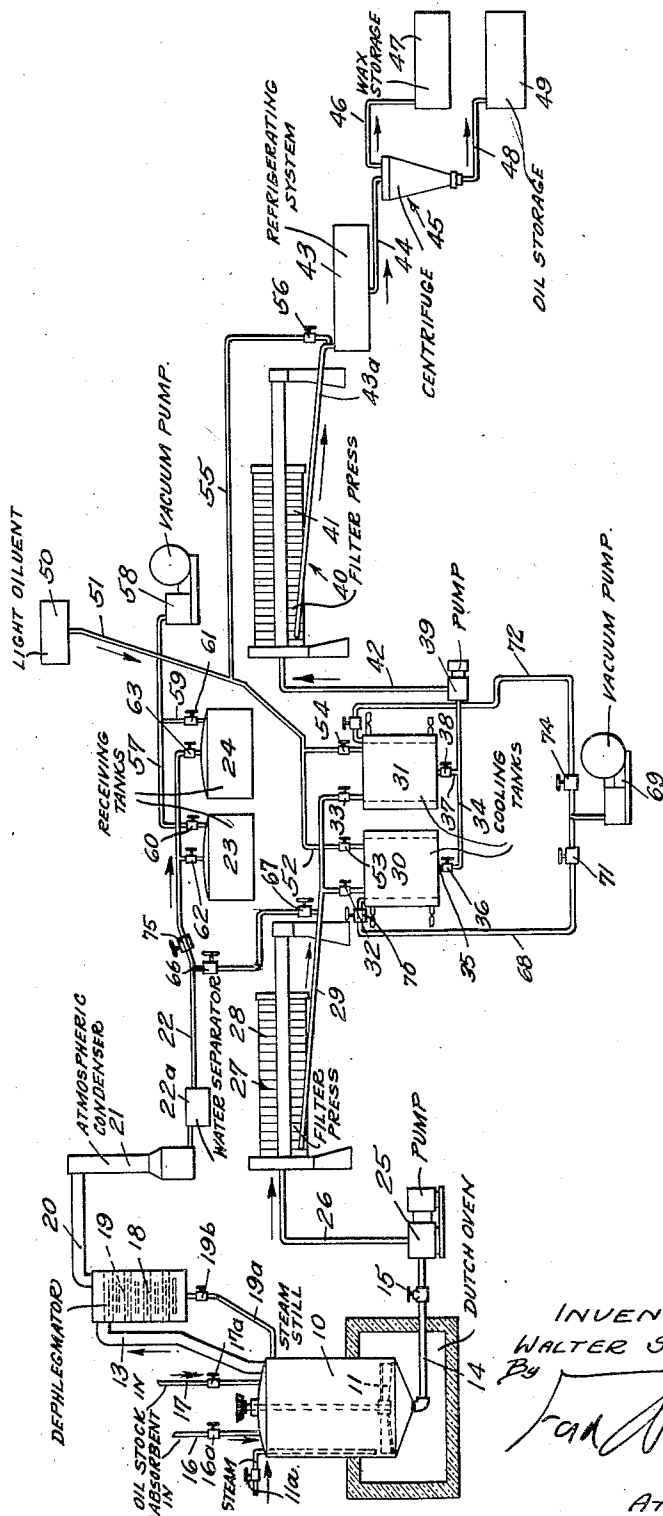


Dec. 19, 1933.

W. S. BAYLIS

1,939,946

PROCESS OF SEPARATING CRYSTALLINE WAX, AMORPHOUS
WAX, AND LUBRICATING OIL FROM WAX SLOP
Filed Feb. 11, 1929



INVENTOR:
WALTER S. BAYLIS,
By *Foran Davis*
ATTORNEY.

UNITED STATES PATENT OFFICE

1,939,946

PROCESS OF SEPARATING CRYSTALLINE WAX, AMORPHOUS WAX, AND LUBRICATING OIL FROM WAX SLOP

Walter S. Baylis, Los Angeles, Calif., assignor to
Filtrol Company of California, Los Angeles,
Calif., a corporation of California

Application February 11, 1929. Serial No. 339,030

9 Claims. (Cl. 196—17)

This invention relates to a process having the object of separating both crystalline and amorphous wax and recovering a maximum yield of lubricating oil from "wax slop". At the present time, a so-called "wax slop" cut is produced during the process of distilling crude petroleum oils in such practice where an intermediate cut between the wax distillate cut and the cylinder stock cut is made. At the present time, there is no known method of treating this stock and separating the wax without cracking, for the reason that the stock contains a mixture of both crystalline and amorphous wax which prevents either cold settling, pressing or centrifuging. This condition is due principally to the presence of so-called "inhibitors" principally of a colloidal nature. By "amorphous wax", I mean solid substances which do not crystallize and are, without definite characteristic shape.

By the practice of my process, I am able to separate colloidal inhibiting and coloring matters, and to so change the character of the wax as to permit it to be readily separated from the stock without any substantial cracking and with practically the theoretical yield of a high-grade lubricating oil of low cold test.

The accompanying drawing shows, more or less diagrammatically, an apparatus suitable for carrying out the process. Other apparatus will serve. In the drawing, 10 indicates a still fitted with an agitator 11 and adapted to serve as a steam still, this still being preferably constructed as a vertical still mounted upon a "Dutch oven". It is provided with a steam inlet pipe 11a, entering the still at the top and being carried down close to the bottom, a vapor outlet pipe 13, and a bottom discharge pipe 14 having a valve 15. An inlet 16 is intended for the introduction of powdered adsorbent, and a pipe 17 is provided to deliver the oil stock to be worked upon.

Above the still 10 the vapor pipe 13 enters a dephlegmator 18, which contains a plurality of interior baffles 19. A pipe 19a connects the bottom of the dephlegmator 18 to the side of the still 10. The pipe 19a contains a valve 19b. A continuation 20 of the vapor pipe 13 is connected to an atmospheric condenser 21. A pipe 22 connects the atmospheric condenser 21 with receiving tanks 23 and 24, respectively, and is provided with a valve 25.

In the pipe 22, between the atmospheric condenser 21 and receiving tanks 23 and 24, is a water-separating device 22a for the purpose of separating condensed steam from the distillate.

A pipe 57 connects the receiving tank 23 to a vacuum-creating device 58 which may be a steam ejector or vacuum pump, said pipe having a branch 59 connecting the receiving tank 24 with the pipe 57. The pipe 57 has a valve 60 and the branch-pipe 59 has a valve 61.

In the pipe 22, just before it enters the receiving tanks 23 and 24, are placed valves 62 and 63. The pipe 14, containing the valve 15, connects the bottom of the still 10 to the suction of a filter-press pump 25. A pipe 26 connects the discharge of the pump 25 to the inlet of a filter press 27 containing filter plates 28. A collecting pipe 29 connects the discharge of the filter press 27 to cooling tanks 30 and 31. This pipe contains valves 32 and 33. The cooling tank 30 is connected to a manifold pipe 34 by means of a pipe 35 containing a valve 36. The cooling tank 31 connects with the manifold pipe 34 by means of a pipe 37 containing a valve 38. The manifold pipe 34 is connected to the suction end of a pump 39. The discharge end of the pump 39 is connected to a filter press 40 containing filter plates 41 by means of a pipe 42. The discharge of the filter press 40 is adapted to be cooled by means of a refrigerating system 43, and is led thereto by a pipe 43a. The entire press may be located in a cold-room or otherwise refrigerated. The refrigerating system 43 is connected by a pipe 44 to the basket of a centrifugal machine 45. The centrifugal machine is connected by a pipe 46 to wax-storage tanks 47, and is also connected by a pipe 48 to storage tanks 49. A tank 50 is a charging tank for a solvent diluent and is connected by means of a pipe 51 to the cooling tank 31. This pipe contains a valve 54. A branch-pipe 52 connects the pipe 51 with the cooling tank 30 and contains a valve 53. A pipe 55 containing a valve 56 is adapted to deliver solvent from pipe 51 to the pipe 43a at a point just before the latter enters the refrigerating system.

A pipe 65 having valves 66 and 67 connects the pipes 22 and 29. A pipe 68 connects the receiving tank 30 to a vacuum creating device 69 which is similar to vacuum creating device 58. The pipe 68 is provided with valves 70 and 71. The pipe 72 connects the vacuum creating device 69 with tank 31 and is provided with valves 73 and 74.

The operation of my process using the above-described apparatus is as follows:

The still 10 is first filled with a suitable charge of the oil stock to be treated through

the pipe 17. To this charge of oil stock is added from two to ten per cent of its weight of powdered mineral adsorbent, preferably of the acid-activated clay type which is introduced into the still through the inlet 16. I prefer to use the kind of acid-activated clay known commercially as "Filtrol", which is an acid-activated clay made by a process somewhat similar to that disclosed in United States Patent to Prutzman, No. 1,397,113, issued November 15, 1921.

The agitator 11 is then started to thoroughly mix the two substances and after a suitable period of mixing, superheated steam at a temperature of from 500° to 700° F. is turned on through the pipe 11a. The valves 15, 16a, and 17a are closed during this operation. Vacuum may or may not be applied, as desired, by means of the pump or ejector 58. The temperature is raised and steam distillation is now carried on until approximately from fifty to sixty per cent by weight of the charge in the still 10 has been distilled overhead and collected in either one of the receiving tanks 23 or 24. Distillation is carried on until the still bottoms have been concentrated to a desired viscosity, the viscosity of these bottoms being dependent upon the character of the raw stock being treated and the kind of oil or wax desired.

The overhead fraction contains light distillates with a large percentage of crystalline wax, and is condensed by means of the air condenser 21 and collected in either of the receiving tanks 23 or 24. This fraction is allowed to stand in storage in the receiving tanks 23 or 24, with or without artificial cooling as desired, for a period of time until the wax has crystallized out. The material is then in condition to be treated by well-known methods, for the removal of the wax in the same manner as wax is removed from a wax distillate cut and the manufacture of light lubricating oils from the residues. After the distillation has been carried to this point it is stopped and valves 16a, 17a, and 15 are opened, the pump 25 started, and the still bottoms of asphaltic, waxy, and adsorbent material remaining in the still 10 are pumped through the pipe 14 by means of the pump 25, and through the pipe 26, into the filter press 27 where the adsorbent material is removed, preferably at 275° F.

The filtrate from the press 27 flows through the pipe 29 into the tanks 30 and 31 which are equipped with means for cooling. The charge of filtrate in either of the tanks 30 or 31 is now diluted with gasoline, distillate, or any other suitable solvent from the tank 50, through the pipe 51, and in the case of tank 30, also through the branch-pipe 52. This dilution is in the ratio of approximately sixty per cent of solvent to forty per cent filtrate. The charge is cooled to between 40° and 60° F. During this cooling operation, crystalline wax with very little, if any, amorphous wax will separate out. When a sufficient length of time has elapsed to permit the wax to form, the wax structure is mechanically broken up and a charge from the cooling tank 30 and/or 31 is discharged through the manifold 34 into the suction end of the pump 39, from whence it is discharged through the pipe 42 into the preferably precooled filter press 40, in which press the crystalline wax is removed.

The filtrate from the press 40 is discharged through the pipe 43a into the refrigerating system 43, in which the mixture of oil and solvent

is cooled to approximately -40° F. It is preferably further diluted by solvent from pipe 55 before it enters the refrigerating system. After cooling to this point, the charge is conducted through the pipe 44 to the centrifugal machine 45, where the amorphous wax is separated, from which it passes through the pipe 46 to the storage tank 47, the de-waxed oil and solvent passing through the pipe 48 to the storage 49. This oil and solvent mixture is then ready to be subjected to distillation for the purpose of driving off the solvent and yielding the lubricating oil as residue. For this purpose well-known standardized types of re-run apparatus may be used.

The oil fraction distilled from the still 10 contains crystalline wax with little or no amorphous wax. The still bottoms from this operation, after filtration to remove the adsorbent material, contain amorphous wax with a very small amount of crystalline wax. This effect of substantially complete removal of the crystalline wax is not obtained without the presence of the adsorbent material in the still during distillation, unless the oil is cracked. Cracking destroys valuable lubricating material and is therefore undesirable here. The rate of distillation affects the separation of crystalline and amorphous wax. Therefore, it is desirable to carry the distillation on as rapidly as possible and at as low a temperature as possible,—hence the desirability of as high a vacuum as possible. Cracking is substantially avoided by the presence of the adsorbent material in the still and by rapid distillation with superheated steam, with or without the aid of reduced pressure.

It lies within the purview of my invention to distill the wax slop completely; that is, to dryness in the still 10 in the presence of the adsorbent. I have found that in such operation no sticky residue, but rather a loose, dry powder is formed. In such distillation, the first fifty per cent to sixty per cent of distillate is run to storage, as usual, to give the crystalline wax time to form, and this wax is separated in the same manner as described in the foregoing matter. The bottom cut is distilled to dryness without breaking the vacuum or interrupting the steam supply, and may be run directly into the tanks 30 and 31 by means of pipes 22, 65 and 29. Before the valves 66 and 67 are opened, and the valve 75 closed, the vacuum creating device 69 is started, and the valves to the proper tank opened. Dilution in these tanks, followed by filter pressing in the press 40, to separate crystalline wax, then occurs. The chilling centrifuging, and solvent separating operations are then practiced in the order named, just as before described. Lighter colored products are produced from the still bottoms of the still 10, when these are completely distilled.

While I prefer to use acid-activated clay in the still 10, I may use naturally active adsorbent clay, or clay which activates easily in the presence of sulphuric acid, even if the clay has no initial decolorizing power. I may use a small percentage of concentrated sulphuric acid in the still 10 during distillation, not to exceed two per cent by weight of the clay used, whether activated or non-activated clay be present. I find that the presence of the acid assists decolorization and tends to improve the quality of the oil.

Instead of acid being present I may substitute small quantities of certain salts resulting from the combination of a weak base and a strong

acid. Examples are aluminum sulphate, magnesium phosphate and manganese phosphate. No more than from 0.25% to 0.50% of such salts, calculated on the weight of the clay, should be present. These salts, I have ascertained, function as activators or catalyzers to fully develop the decolorizing action obtainable from the clays.

In the foregoing matter and in the appended claims, the word "bottoms" means the heavy asphaltic, waxy, and adsorbent-containing residue of distillation of about 50% by weight of the wax slop, while the word "distillate" means the total fractions distilled over, these consisting of lubricating oil and crystalline wax.

I claim as my invention:

1. A process of separating crystalline wax, amorphous wax and lubricating oil from wax slop which comprises: steam distilling a mixture of adsorbent clay and wax slop in which there is a small percentage of an activating substance which promotes adsorption until approximately from fifty to sixty per cent by weight of the charge has been distilled over, slightly chilling the distilled fraction and separating the wax therefrom, separating the clay from the bottoms of the distillation, diluting the bottoms with a light hydrocarbon, chilling the diluted bottoms to a low temperature to separate the crystalline wax, filtering the separated wax from the filtrate, chilling the filtrate to a lower temperature, separating the amorphous wax and removing the light hydrocarbon from the oil after the wax has been separated therefrom.

2. In a process of separating crystalline wax, amorphous wax and lubricating oil from wax slop the steps which comprise: diluting the bottoms of a steam distillation of said wax slop in the presence of an adsorbent clay with a light hydrocarbon, chilling the diluted bottoms to a low temperature separating the crystalline wax, filtering the separated wax from the clear bottoms, chilling the clear bottoms to a lower temperature, and separating the amorphous wax.

3. In a process of separating crystalline wax, amorphous wax and lubricating oil from wax slop the steps which comprise: diluting the bottoms of a steam distillation in vacuo of said wax slop in the presence of an adsorbent clay with a light hydrocarbon, chilling the diluted bottoms to a low temperature separating the crystalline wax, filtering the separated wax from the clear bottoms, and chilling the clear bottoms to a lower temperature to separate the amorphous wax.

4. A process of separating crystalline wax, amorphous wax and lubricating oil from wax slop which comprises: steam distilling a mixture of adsorbent clay and wax slop, the clay

being present in the proportion of two to ten per cent of the slop, until approximately fifty per cent by weight of the charge has been distilled over, diluting the filtered bottoms with a substantial proportion of light hydrocarbon, cooling the mixture containing the filtered bottoms to approximately from 30° F. to 60° F., filtering the mixture containing the filtered bottoms to separate the crystalline wax, refrigerating the filtrate to a temperature sufficiently low to chill out substantially all of the remaining wax, separating the wax, and then recovering the lubricating oil.

5. A process according to claim 4 in which the adsorbent clay is a finely divided acid activated clay containing free acid.

6. A process of separating crystalline wax, amorphous wax and lubricating oil from wax slop which comprises: steam distilling a mixture of adsorbent clay and wax slop, the clay being present in the proportion of two to ten per cent of the slop, until approximately fifty per cent by weight of the charge has been distilled over, diluting the filtered bottoms with a substantial proportion of light hydrocarbon, cooling the mixture to approximately from 30° F. to 60° F., filtering the mixture to separate the crystalline wax, refrigerating the filtrate to approximately -50° F. to chill out substantially all of the remaining wax, separating the wax, and recovering the lubricating oil from the light hydrocarbons.

7. In a process of separating crystalline wax, amorphous wax and lubricating oil from a wax slop the steps which comprise: steam distilling a mixture of adsorbent clay and said wax slop, separating the crystalline wax and amorphous wax from the last forty to sixty per cent of the said mixture by diluting said portion with a light petroleum oil, chilling the mixture to about 40° F. to separate the crystalline wax, filtering the separated wax from the mixture, refrigerating the filtrate to about -50° F. separating the amorphous wax, and recovering the lubricating oil from the light petroleum oil after the wax has been separated therefrom.

8. A process according to claim 7 in which the adsorbent clay is an acid-activated clay which contains a small percentage of an adsorption promoting substance.

9. A process according to claim 7 in which the distillation is carried on as rapidly as possible and at as low a temperature as possible in order to obtain a good separation of crystalline and amorphous wax.

WALTER S. BAYLIS.