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SOLVENT EXTRACTION OF WAXY OILS

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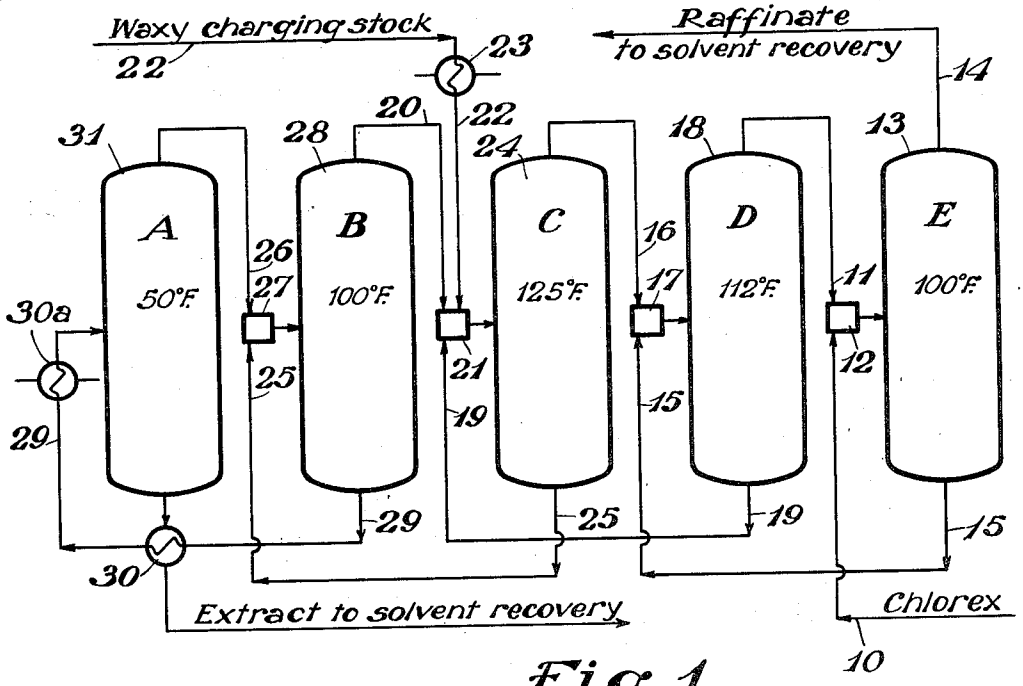

Fig. 1

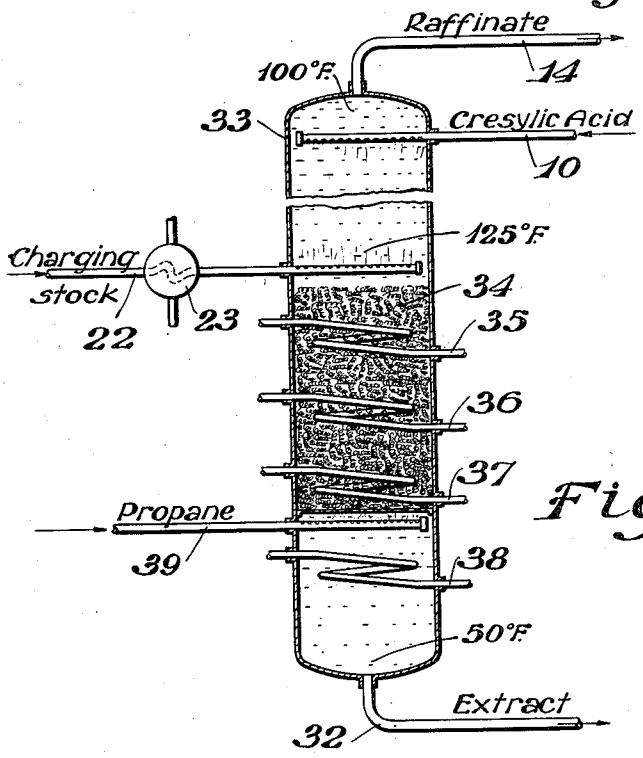

Fig. 2

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SOLVENT EXTRACTION OF WAXY OILS

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8 Claims. (Cl. 196—13)

This invention relates to improved methods and systems for the solvent extraction of waxy oil stocks and it pertains more particularly to the refining of wax bearing petroleum oils for the separation of paraffinic from naphthenic components by means of solvents such as dichlorethyl ether which operate at temperatures between about 50 and 125° F. Dichlorethyl ether is commonly known in the art as Chlorex and will hereinafter be referred to as such.

The most desirable operating temperatures for solvent extraction systems are those which do not require excessive amounts of heating or refrigeration or, in other words, are temperatures of about 50 to 125° F. Many of the most effective and most satisfactory solvents, such as Chlorex, aqueous phenol and cresylic acid, chlorophenol mixtures, nitro benzene, etc. operate in this general temperature range. It has been found, however, that when the lubricating oil stocks contain appreciable amounts of wax, particularly crystalline wax, the undewaxed stock is difficult to handle in this temperature range because the solidification of the wax results in a soupy mixture or buttery mass which is extremely hard to handle in ordinary mixers and settlers and which it is practically impossible to separate from solvent extract mixtures without the use of centrifuges.

The object of my invention is to provide methods and means for solvent extracting such wax bearing oils in ordinary settlers or countercurrent towers.

A further object of my invention is to increase the yield of paraffinic oils obtainable from any given stock or, in other words, to prevent the loss of paraffinic oils with extract material. A further object is to provide a system for solvent extracting waxy oils in a packed countercurrent tower by means of solvents such as Chlorex and chlorophenol without encountering difficulties due to solidification of wax and entrainment of solvent extract material by said wax. Other objects will be apparent as the detailed description of my invention proceeds.

In practicing my invention I employ a countercurrent system which may be either a packed tower or a multiple batch countercurrent system and I introduce the waxy oil stock at an intermediate point in the system and I maintain the temperature at this intermediate point higher than the temperatures on both sides of this point. For instance, in a packed countercurrent Chlorex tower I introduce waxy oil at a temperature of about 125° F. at an intermediate and preferably relatively high point in the tower (above the packed portion). The Chlorex is introduced at the top of the tower and the temperature gradient in the upper part of the tower may be gradually decreased toward the raffinate exit point due to heat losses due to radiation and due to the lower temperature of the incoming Chlorex. Practically all of the wax remains in the upper part of the tower and it therefore does not clog the packing material in the lower part of the tower wherein it is essential that good contact be maintained between liberated or undissolved paraffinic oils and solvent. I maintain the extract exit at a relatively low temperature, preferably about 50° F., since I have discovered that it is the extract exit temperature which, in effect, prevents the loss of paraffinic oils from the system and therefore insures maximum yields of high quality lubricating oils.

The invention will be more clearly understood by reference to the accompanying drawing wherein similar parts are designated by like reference characters in the two figures and wherein:

Figure 1 is a diagrammatic elevational plan of a multibatch countercurrent extraction system; and

Figure 2 is a vertical section of a packed countercurrent extraction tower diagrammatically illustrating the invention.

Any solvent may be used in practicing my invention which has an operating range within about 0 to 150° F. or preferably within about 50 to 125° F. Preferred examples are Chlorex, ortho monochlorophenol and chlorophenol mixtures, aqueous chlorophenol, aqueous phenol, aqueous cresylic acid or aqueous phenol-cresylic acid mixtures, nitro-benzol, etc. The invention will be described in connection with Chlorex systems. The oil to be treated may be a distillate or residual stock which may or may not have undergone previous treatments to remove asphalts, resins or other undesirable materials. For instance, the stock may have undergone precipitation with propane for removal of asphalts and resins, it may have been subjected to a light acid treatment or, particularly in the case of extremely naphthenic oils, it may have undergone a preliminary solvent extraction treatment to remove extremely low grade materials. In the preferred example I will describe the extraction of a Midcontinent distillate stock having a viscosity of about 70 seconds at 210° F., but it should be understood that the invention is equally applicable to other distillates, other crudes and other conditions.

Referring to Figure 1, a multistage extraction system is indicated as stages A, B, C, D and E, beginning with the stage of extract removal, although it should be understood that any number of stages may be used. Chlorex is introduced through line 10 and admixed with raffinate material from stage D from line 11 in mixer 12, the mixture being introduced at the middle of settler 13 in stage E. The final raffinate from this stage is removed through line 14. An extract material from stage E is returned through line 15 and admixed with raffinate material from stage C from line 16 in mixer 17, this mixture being introduced at the center of settler 18 in stage D. Extract material from this stage is returned through line 19 for admixture with raffinate material from stage B which is introduced through line 22 and heater 23, the temperature of the heater being sufficient to maintain the temperature of the entire mixture in the following stage at about 125° F. The solvent extract material from line 19 is intimately mixed with raffinate material from line 20 and incoming stock from line 22 in mixer 21 and this mixture is introduced into the middle of settler 24 in stage C.

Extract from this settler is withdrawn through line 25 and mixed with raffinate material from stage A which is introduced through line 26 into mixer 27, the mixture being introduced into the middle of settler 28 of stage B.

The extract material from stage B, which is preferably maintained at a temperature of about 100° F., is withdrawn through line 29, exchanger 30 and cooler 30a and introduced at the center of settler 31 in stage A, final extract material being withdrawn through line 32 to the extract solvent recovery system.

It will be noted that the extract entering stage A is not mixed with any other material but is merely cooled to a low temperature to effect the separation in this stage which is preferably maintained at about 50° F. or lower. I have found that the temperature of the extract exit stage determines the selectivity of the extraction. The temperature at the raffinate end of the system may be considerably higher but there will be no loss of selectivity in the system as a whole if the temperature at the extract end of the system is maintained sufficiently low (provided, of course, that adequate means are provided for the separation of paraffinic oil from solvent in this low temperature zone). Other conditions being equal, the lower the temperature of extraction at the extract end, the better will be the selectivity.

It will be noted that the wax will be concentrated in my improved system in stages C, D and E which are all operating at relatively high temperatures. The wax which meets incoming solvent in stage E is substantially freed from asphaltic and naphthenic materials and therefore causes no entrainment or separation problems. If this wax were present in stages A and B there would be mechanical entrainment of phases between stages with resulting lowering of effectiveness of the stage compared with the theoretical stage.

As indicated in the drawing, a preferred range of operating temperatures is 50°, 100°, 125°, 112° and 100°, respectively, for stages A, B, C, D and E. It should be understood, however, that these temperatures are merely illustrative; that temperatures as high as 150° may be used in stage C; that the temperature in stages C, D and E may be substantially constant; and that temperatures as high as 75° or as low as 0° F. may be used in stage A. In the case of low miscibility temperatures solvents such as nitro benzene refrigeration need be applied only to the extract exit end of the system. Suitable heat exchange may be employed throughout the various parts of the system as will be apparent to those skilled in the art.

My invention is of still greater significance in systems employing packed countercurrent extraction towers since it is practically impossible to countercurrently extract a soupy wax slurry in conventional equipment. In accordance with my invention the incoming stock through line 22 is passed through heater 23 so that the wax-oil solution will be mobile and fluid and at a temperature of about 120 to 150° F. This mixture is introduced into countercurrent tower 33 above the packed portion thereof. Under conditions at this point in the tower there will be a substantially complete separation of the wax from naphthenic materials, the wax being carried upwardly with the raffinate and the naphthenic materials with entrained naphthenic oil being carried downwardly through the packed section of the tower. Chlorex or other solvent may be introduced at the top of the tower through line 10 and raffinate may be removed through line 14, the top of the tower being maintained at a temperature of about 100° F.

The packing material 34 in the lower part of the tower may be of any conventional type of material such as graphite, Raschig rings, glass beads, porcelain figures, steel jack chain or steel turnings. I have found that the steel turnings or steel springs give excellent contact in this portion of the system but I do not limit myself to any particular type of packing material.

The temperature gradient in the lower part of the tower is gradually decreased from the point of charging stock introduction to the extract removal through line 32. This lowering of temperature is preferably effected by a series of pancake coils 35, 36, 37 and 38 so that at the bottom of the tower the temperature is about 50° although it may be as high as 75 and as low as 0° F.

As the mixture is cooled in the lower part of this packed tower paraffinic oils are released from the solvent extract solution and the release of such oils is materially facilitated and expedited by the use of the packing material. Ordinarily it is unnecessary to introduce any stripping or washing fluid at the base of the tower but in some cases such a material (for instance propane) may be introduced through line 39 and passed upwardly through the packed section of the tower.

The propane tends to wash the extract and to cause further separation of paraffinic oil from solvent extract material. Other light hydrocarbon diluents may also be employed for this purpose. I prefer to avoid the use of light oil diluents in a Chlorex extraction system but I have found that the use of propane or other light hydrocarbon diluents may be extremely effective in systems employing phenol, cresylic acid, chlorophenol, etc.

In the above example the proportions of solvent to oil are usually about two to one, although these proportions may vary from one to one—to three to one and the exact diluent ratio must be determined in every case by the nature of available stock and the specifications of the desired product.

By operating all but the extract stages at high temperature I effect a considerable saving in refrigeration and by eliminating the wax from

the extract material before refrigeration is employed I increase the effectiveness and efficiency of refrigeration. The saving in refrigeration, together with the increased selectivity of extraction and the increased ease of separating raffinate from extract material makes my improved process one of great commercial importance in the refining of wax bearing lubricating oil stocks.

I claim:

1. The method of countercurrently extracting a waxy oil stock in a packed tower which comprises introducing the waxy oil charging stock at a point in the tower above the packing material and at a temperature at which the waxy oil is fluid and mobile, introducing solvent at the top of said tower and flowing downwardly in said tower countercurrent to the upward flow of paraffinic and waxy materials, withdrawing extract from the base of said tower and cooling the extract material in the lower part of the tower whereby the paraffinic oil is released from the extract solution and the downcoming extract solution is contacted with said released paraffinic oil in the packed section of the tower.

2. The method of claim 1 wherein the temperature in the base of the tower is from 0° to 50° F.

3. The method of claim 1 wherein dichlorethyl ether is the extracting solvent and the temperature at the base of the tower is about 50° F.

4. The method of claim 1 which also includes the introduction at the base of the tower of a light stripping liquid for facilitating the removal of paraffinic oil from extract material in the packed section of the tower.

5. The method of claim 1 wherein propane is introduced in the lower part of the tower.

6. The method of countercurrently extracting a waxy oil stock in a packed tower which comprises introducing said stock at a point in the tower above the packing material and at a temperature of at least 100° F., introducing solvent at the top of said tower and countercurrently contacting said stock with said solvent in the upper part of said tower at a temperature at which the wax is in liquid phase, effecting a separation of paraffinic oil from the extract in the lower part of said tower and contacting said separated oil with downcoming extract in the packed section of the tower.

7. The method of claim 6 wherein the separation in the lower part of the tower is effected by chilling to a temperature not higher than 50° F.

8. The method of claim 6 wherein the separation in the lower part of the tower is effected by a modification of the solvent caused by the introduction of a liquid to bring about a separation of paraffinic oil from extract.

ARTHUR B. BROWN.