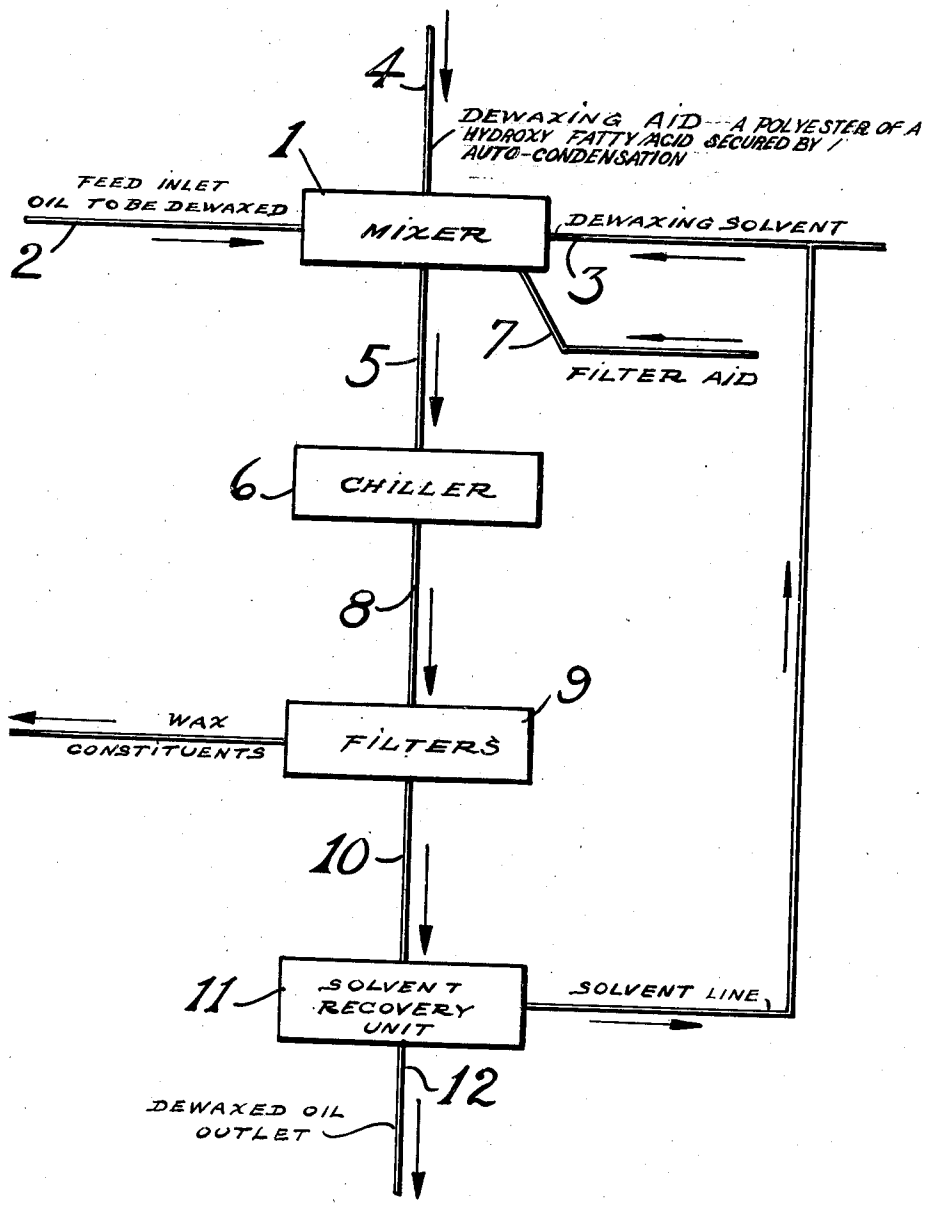
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METHOD OF DEWAXING MINERAL OIL

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METHOD OF DEWAXING MINERAL OIL

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10 Claims. (Cl. 196—18)

The present invention relates to an improved process for producing oils of low cold test from oils which contain appreciable amounts of waxy hydrocarbons. The invention particularly relates to a process for producing lubricating oils by an improved method of dewaxing wax-bearing mineral oils. The invention especially relates to the utilizing as dewaxing and filtering aids an ester of a hydroxy fatty acid, particularly polyesters of hydroxy fatty acids which are of high molecular weight and viscosity.

It is known to utilize various wax crystal modifying agents in order to aid the removal of both amorphous and crystalline wax. These agents are used in the various processes of dewaxing oil as, for example, plate and frame pressing, cold settling and centrifuging. The agents of the present invention are particularly desirable for the removal of amorphous and semi-crystalline wax, which are usually removed by cold settling or centrifuging. These agents, however, are also useful for increasing filtering rates in the plate and frame or similar process.

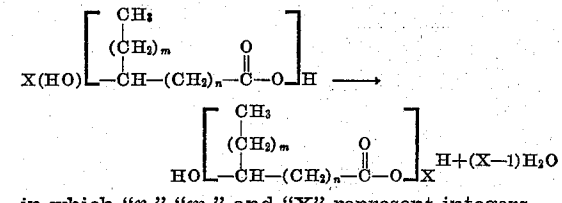
Waxy mineral oils are usually dewaxed to produce oils of low cold test by diluting with some solvent, which may be hydrocarbon or non-hydrocarbon in character, and the diluted solution then chilled until the wax crystallizes out. The cold oil is then usually held at the dewaxing temperature until the wax has settled out either at the top or bottom, depending upon the specific gravity of the particular solvent used.

It has now been found that the separation of the wax can be greatly accelerated by adding the dewaxing aids of the present invention to the solution during the dewaxing operation. These materials apparently collect on the wax crystals, modifying the same and preventing them from being wet by the oil. Under such conditions the wax separates quickly and cleanly from the oil solution.

The present invention may be readily understood by reference to the drawing which demonstrates one suitable modification of the same. In this process the oil to be dewaxed is led into mixer 1 by means of feed line 2. A suitable dewaxing solvent is led into mixer 1 by means of feed line 3 while the dewaxing aid of the present invention is led in by means of feed line 4. Filter aids may also be incorporated if desired by means of line 7. After thorough mixture, preferably at about room temperature, the material is passed through line 5 into chiller 6 where it is chilled to the desired temperature, usually in the range from minus 30° F. to plus 30° F., preferably from 0° F. to 20° F. and allowed to stand until the desired wax separation occurs. These conditions may, of course, be varied in order to produce any particular low pour point oil. The chilled mixture is then led through line 8 through the filters 9 where the waxy constituents are separated from the oil. The wax is removed and the dewaxed oil and solvent are then led through line 10 into the solvent recovery unit 11 wherein the solvent is recovered from the dewaxed oil which is removed through line 12.

The dewaxing and filtering aids of the present invention are fatty acid esters of high molecular weight hydroxy fatty acids which are soluble or at least colloidally dispersed in common dewaxing solvents as, for example, a hydroxy stearic acid which has been esterified with stearic acid or with another fatty acid as, for example, palmitic acid. The preferred materials of the present invention are high molecular weight polyesters which have been prepared by the auto-condensation of a hydroxy fatty acid. These latter materials are preferably prepared from monohydroxy acids and have molecular weights above 1,500, preferably in the range from 3,000 to 10,000 and higher and a viscosity Saybolt at 100° F. of at least 50,000 seconds.

The production of the high molecular weight polyesters of the present invention may be illustrated by the following formula:



in which "*n*," "*m*," and "X" represent integers.

The high molecular weight polyesters may be manufactured in any manner whatsoever. For example, castor oil may be hydrogenated in the presence of platinum or a nickel catalyst at from 5 to 10 pounds pressure to form hydroxy stearic acid and the polyester then formed by auto-esterifying the hydroxy stearic acid under suitable auto-esterifying conditions.

For example, preferred polyesters may be secured by auto-esterifying organic hydroxy acids having more than 5 or 6 carbon atoms between the hydroxyl and carboxyl groups. The polyesters may be formed from the hydroxy acid by utilizing heat in the presence of catalyst as, for example, sulphuric acid. They are preferably prepared by utilization of catalysts as, for example, chloracetic, dichloracetic, fumaric, phthalic and adipic acids at a temperature in the range from 212° to 575° F., preferably at a temperature from 390° to 430° F. These polyesters are treated under these conditions for approximately 10 to 150 hours, depending upon the molecular weight desired.

These dewaxing and filtering aids may be added in any concentration desirable, depending upon the type of material being dewaxed, the tempera-

ture of dewaxing, the solvent being used and the pour point desired in the dewaxed product. The materials are usually added in the amount from 0.02% to 5%, preferably in the concentration from 0.2% to 1%.

The solvents employed in the present process may be any suitable solvent as, for example, benzol, acetone, toluol, methyl ethyl ketone, propane, ethylene dichloride, aliphatic alcohols, naphtha, and the like. These solvents may be combined with various other particular solvents in various concentrations to secure optimum results. Particularly desirable results are secured with the settling and filtering acids of the present invention when a naphtha solvent is employed.

The following specific examples are given in order to demonstrate the invention and are not to be construed as limiting the same in any manner whatsoever.

Example 1

Mono 12 hydroxy stearic acid was prepared by hydrogenation and saponification of castor oil which was purified by separating the excess stearic acid by extraction with benzine. This material was heated in an inert atmosphere for several days at a temperature of from about 390° to 430° F. with stirring. The resultant product had an average molecular weight of about 5,000, a neutralization value of about 10.1 mg. KOH/gm. and a viscosity well above 50,000 seconds Saybolt at 100° F. Approximately 0.5% of this material was then added to a waxy lubricating oil and the settling rate of the blended and of the unblended oil was determined at 0° F. The results of these tests were as follows:

	At the end of minutes			
	15	30	45	60
Waxy oil alone:				
Volume clear oil	1	1	1	1
Volume waxy oil	99	99	99	99
Waxy oil plus 0.5% settling aid:				
Volume clear oil	25	40	65	78
Volume waxy oil	75	60	35	22

Example 2

A Reagan oil distillate having a 72 viscosity at 210° F. was diluted with naphtha having a gravity A. P. I. of 54°. To this material was added 0.5% of a polyester of hydroxy stearic acid prepared in the above described manner and the settling rates determined at 0° F. on the oil containing the polyester as compared to the blend not containing the polyester. The results of these tests were as follows:

	At the end of hours			
	¼	½	¾	1
20 volumes Reagan distillate, 80 volumes 54° naphtha:				
Volume clear oil	0	0	0	0
Volume waxy oil	100	100	100	100
20 volumes Reagan distillate; 80 volumes 54° naphtha, 0.5% polymer of hydroxy stearic acid:				
Volume clear oil	28	43	68	78
Volume waxy oil	72	57	32	22

The above invention is not to be limited by any theory or method of operation but only by the following claims in which it is desired to claim all novelty in so far as the prior art permits.

We claim:

1. Improved method of dewaxing a mineral oil comprising mixing with the said oil a polyester of a hydroxy fatty acid secured by auto-condensation of a high molecular weight mono-hydroxy fatty acid, chilling the oil and separating the precipitated wax.

2. Process in accordance with claim 1 in which said mono-hydroxy fatty acid is mono-hydroxy stearic acid.

3. Improved method of dewaxing a waxy petroleum oil comprising mixing with the said oil a polyester of a hydroxy fatty acid secured by auto-condensation of a high molecular weight mono-hydroxy fatty acid, said ester having a molecular weight of at least 3,000 and a viscosity Saybolt of at least 50,000 seconds at 100° F., chilling the oil and separating the precipitated wax.

4. Improved method of dewaxing a wax-bearing petroleum oil comprising mixing with the said oil a polyester secured by auto-condensation of a high molecular weight mono-hydroxy fatty acid, said polyester having a molecular weight of at least 3,000 and a viscosity Saybolt of at least about 50,000 seconds at 100° F., chilling the oil and separating the precipitated wax.

5. Process in accordance with claim 4 in which said polyester is secured by auto-condensation of a mono-hydroxy stearic acid.

6. Process for dewaxing a wax-bearing petroleum oil comprising mixing with the said oil a dewaxing solvent and a polyester of a mono-hydroxy mono-basic fatty acid having a molecular weight of at least 1,500, and formed by auto-esterifying the said mono-hydroxy mono-basic fatty acid, chilling the oil and separating the precipitated wax.

7. Process for dewaxing a wax-bearing petroleum oil comprising mixing with the said oil a dewaxing solvent and an auto-esterified polyester of mono-hydroxy mono-basic fatty acids, chilling the oil and separating the precipitated wax.

8. Process in accordance with claim 7 in which said dewaxing solvent is naphtha.

9. Process for dewaxing a wax-bearing petroleum oil comprising mixing with the said oil a dewaxing naphtha solvent and an auto-esterified polyester of a hydroxy long chain fatty acid, said polyester having a molecular weight of at least 3,000 and a viscosity Saybolt of at least about 50,000 seconds at 100° F., chilling the oil and separating the precipitated wax.

10. Process in accordance with claim 9 in which said polyester is secured by auto-condensation of mono-hydroxy stearic acid.

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