United States Patent Office 2,731,391
Patented Jan. 17, 1956

2,731,391

CLOUD POINT REDUCTION OF MINERAL WHITE OILS

Leo Salzmann, Petrolia, Pa., assignor to L. Sonneborn Sons, Inc., a corporation of Delaware

No Drawing. Application September 19, 1950, Serial No. 185,693

10 Claims. (Cl. 196—17)

This invention relates to new and useful improvements in the cloud point reduction of mineral white oils.

Mineral white oils derived from wax bearing petroleum oil fractions usually possess a relatively high cloud point, i. e., that temperature at which the higher melting wax fractions begin to crystallize in the mineral white oil forming a noticeable haze therein. These oils also possess a pour point, i. e., the temperature point at which the oil loses fluidity.

When the temperature of these oils drops to the cloud point, the oils become cloudy, and they will not become clear again until they are subjected to temperatures greatly in excess of the temperatures that they are normally subjected to. These mineral white oils are marketed as a water clear product, and any cloudiness that they might possess would interfere with their marketability. For this reason, it is highly desirable to lower the cloud point to a temperature that these oils, in their normal use and distribution, would not encounter. Since the pour points of these mineral white oils are usually relatively low, their reductions are in many cases relatively less important.

Co-pending application Serial Number 185,691, of even filing date herewith, shows the pour point and cloud point reduction of petroleum oil fractions. As there disclosed, petroleum oil fractions are brought into intimate contact with urea at a temperature of about 60–110° F., in the presence of a suitable activator. The urea takes up waxy or paraffinic constituents of the petroleum oil fractions. By this method, the pour point and the cloud point of the oil fraction are reduced. This procedure may be carried out in batch or multiple stage treatments.

When the method described in said copending application is used on a naphthenic mineral white oil, containing less than 1% paraffin wax, no appreciable reduction of the cloud or pour point is obtained.

One object of this invention is the reduction of the cloud and pour points of a mineral white oil containing less than 1% paraffin wax. These and other objectives will become clear from the following description.

In accordance with the invention, the cloud and pour point of a mineral white oil is reduced by intimately contacting it with an adsorbent carrying dispersed therethrough urea and preferably in addition an activator.

The cloud point reduction may be conducted either in batch treatment or by passing the white oil through the urea carrying adsorbent.

The mineral white oil to be treated in accordance with the invention is a conventional petroleum oil fraction containing some wax and obtained in the well-known exhaustive fuming sulfuric acid refining of a petroleum oil distillate.

The adsorbent useful for the invention may be any adsorbent, preferably of the "activated" type, conventionally known and used in the petroleum industry. Suitable adsorbents of this kind are, for instance, fuller's earth, silica gel, activated bauxite and similar materials.

The activator useful in the preferred application of the invention is preferably at least one lower alcohol not exceeding about 3 carbon atoms in the aliphatic chain. Satisfactory activators, for instance, are methanol, ethanol, propyl and isopropyl alcohol. Methanol, however, gives particularly good results, and it is the preferred activator material.

It has been found that at least 2.5% urea by weight of the urea-carrying adsorbent should be present and preferably 5% to 15% urea by weight of the urea-carrying adsorbent be present in accordance with the invention.

The mesh sizes of the adsorbent should be those conventionally used for decolorization in the petroleum industry.

The adsorbent is mixed with the urea preferably in the presence of the activator, the resulting urea carrying adsorbent is then brought into intimate contact with the white oil for cloud point reduction thereof.

The adsorbent-urea composition may be formed by treating the adsorbent with a solution of urea prepared for instance by dissolving or slurrying the urea in the activator. The adsorbent composition is then dried until the activator concentration does not substantially exceed 4%. This composition is then brought into intimate contact with a mineral white oil either by mixing the material with the white oil, or passing the white oil through the composition.

The urea adsorbent composition may also be made by grinding the urea to a fine powder and then mixing it with the adsorbent which has been wetted with the activator.

While it has been found that the presence of at least one mol of the activator per 2 mols of the urea carried by the adsorbent is necessary to obtain the maximum cloud point reduction, partial cloud point reduction may be obtained without the presence of any activator.

Though the depression of cloud point is normally the primary consideration in the treatment in accordance with the invention, it has been found that the same is also effective to reduce the pour point of a mineral white oil.

EXAMPLE 1

Regular reburnt bauxite was screened from fines, activated by heating to 700° F. and after cooling immersed in an excess of a saturated solution of urea in methanol to form a slurry. After standing for an hour, the slurry was quickly freed from excess solution by filtering over a Buechner filter. Portions of the wet filter residue were dried in a vacuum oven at 180° F. for different lengths of time. A batch of naphthenic mineral white oil having the following characteristics: viscosity at 100° F. 355 Saybolt Universal seconds, pour point −10° F., cloud point +38° F., flash point 430° F., specific gravity .8850 was used, in this experiment. 250 cc. of this white oil was percolated over 40 cc. portions of each composition sample. The urea content of the bauxite urea methanol composition was determined by Soxhlet extraction of a 40 gram sample with methanol over a period of 6 hours and was found to be about 8.0%. The methanol content of the bauxite urea methanol combination was determined by drying 10–12 gram portions in the vacuum at 180° F. overnight. The results are given in Table I. Table I shows also the cloud point of the filtrate of the white oil.

Table I

Drying Time	Wt. Percent Methanol	Cloud Point of Filtrate
60 min.	4.99	−2
75 min.	4.95	−4
90 min.	2.94	−4
105 min.	1.34	+22
120 min.	.98	+28
180 min.	.16	+24
Bauxite alone		+38

EXAMPLE 2

Various proportions of activated bauxite, obtained as in Example 1, of a mesh size conventionally used for decolorization, and urea-bauxite composition as shown in Table II below, were brought in intimate contact with various batches of the same oil used in Example 1. This contact was accomplished by percolating the mineral white oil through the bauxite-urea composition using quantities of 250 cc. of oil to 40 cc. of the bauxite urea material. The reduction in the cloud and pour point of the oil as shown against various percentages of the activated bauxite and the urea is shown in Table II below.

Table II

FILTER MATERIAL

Percent Reg. Bauxite	Percent Urea-Bauxite Composition	Cloud Point	Pour Point
100	0	+38	−10
75	25	+12	−15
50	50	0	−25
0	100	− 4	−25

EXAMPLE 3

Regular bauxite was thoroughly mixed with finely powdered urea. After charging filter tubes with 40 cc. of this dry mixture, the filter material was wetted with methanol, and 250 cc. of the same white oil as used in Example 1 was percolated over the filter bed. Table III below shows the relation of the per cent of dry urea and the volume of methanol to the cloud point of the filtrate.

Table III

Wt. Percent dry Urea Added	Vol. Percent Methanol Added (on clay)	Cloud Point of Filtrate
none	none	+38
10	none	+24
10	5	+ 8
10	10	+12
2.5	5	+22

The above examples were repeated, using in turn fuller's earth and then silica gel in place of the bauxite. Comparable results were obtained.

The above examples are given by way of illustration only and are in no way intended to limit the scope of the invention. The scope of the invention is limited by the appended claims or their equivalents.

I claim:

1. Method for the reduction of the normally relatively high cloud point of a mineral white oil, the cloud point of which is due to the less than 1% of wax contained therein, which comprises intimately contacting such white oil with an adsorbent carrying dispersed therethrough urea, at least a portion of which is in adsorbed condition resulting from alcoholic urea solution contact, and recovering mineral white oil of lower cloud point, said urea-carrying adsorbent being present in amount sufficient to lower said cloud point at least 15° F.

2. Method in accordance with claim 1 in which there is present at least 2.5% urea by weight of the total urea-carrying adsorbent present.

3. Method in accordance with claim 1 in which said urea is present in amount from 5% to 15% by weight of the total urea-carrying adsorbent present.

4. Method in accordance with claim 1 in which said urea-carrying adsorbent is obtained by mixing said adsorbent and at least 2.5% urea by weight of the total urea and adsorbent present in the presence of an activator, being at least one aliphatic alcohol having not over 3 carbon atoms.

5. Method in accordance with claim 4 in which said adsorbent is at least one member of the group consisting of fuller's earth, silica gel, and bauxite.

6. Method in accordance with claim 4 in which said adsorbent is activated bauxite.

7. Method in accordance with claim 1, in which said urea-carrying adsorbent is obtained by substantially dissolving urea in at least one aliphatic alcohol not having over 3 carbon atoms and mixing said adsorbent with said urea solution and removing said alcohol from said mixture until the alcoholic content thereof does not substantially exceed 4%.

8. Method in accordance with claim 1 in which said urea-carrying adsorbent is obtained by grinding urea to a fine powder and mixing the said powdered urea with the adsorbent which has been wetted with an aliphatic alcohol having not over 3 carbon atoms, said urea being present in at least 2.5% by weight of the weight of the urea-carrying adsorbent.

9. Method in accordance with claim 1 where said intimate contact is accomplished in the presence of an activator, said activator being at least one member of the group consisting of methanol, ethanol, propyl and isopropyl alcohol, the amount of said activator being at least one mol per 2 mols of the said urea-carrying adsorbent, said urea being present in amount of 2.5% by weight of the total weight of the urea-carrying adsorbent, said adsorbent being at least one member of the group consisting of fuller's earth, silica gel, and bauxite.

10. Method in accordance with claim 1 in which said intimate contact is accomplished by percolating said white oil over the said urea-carrying adsorbent, said urea-carrying adsorbent containing an activator being at least one aliphatic alcohol having not over 3 carbon atoms, said adsorbent being at least one member of the group consisting of fuller's earth, silica gel, and bauxite.

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