

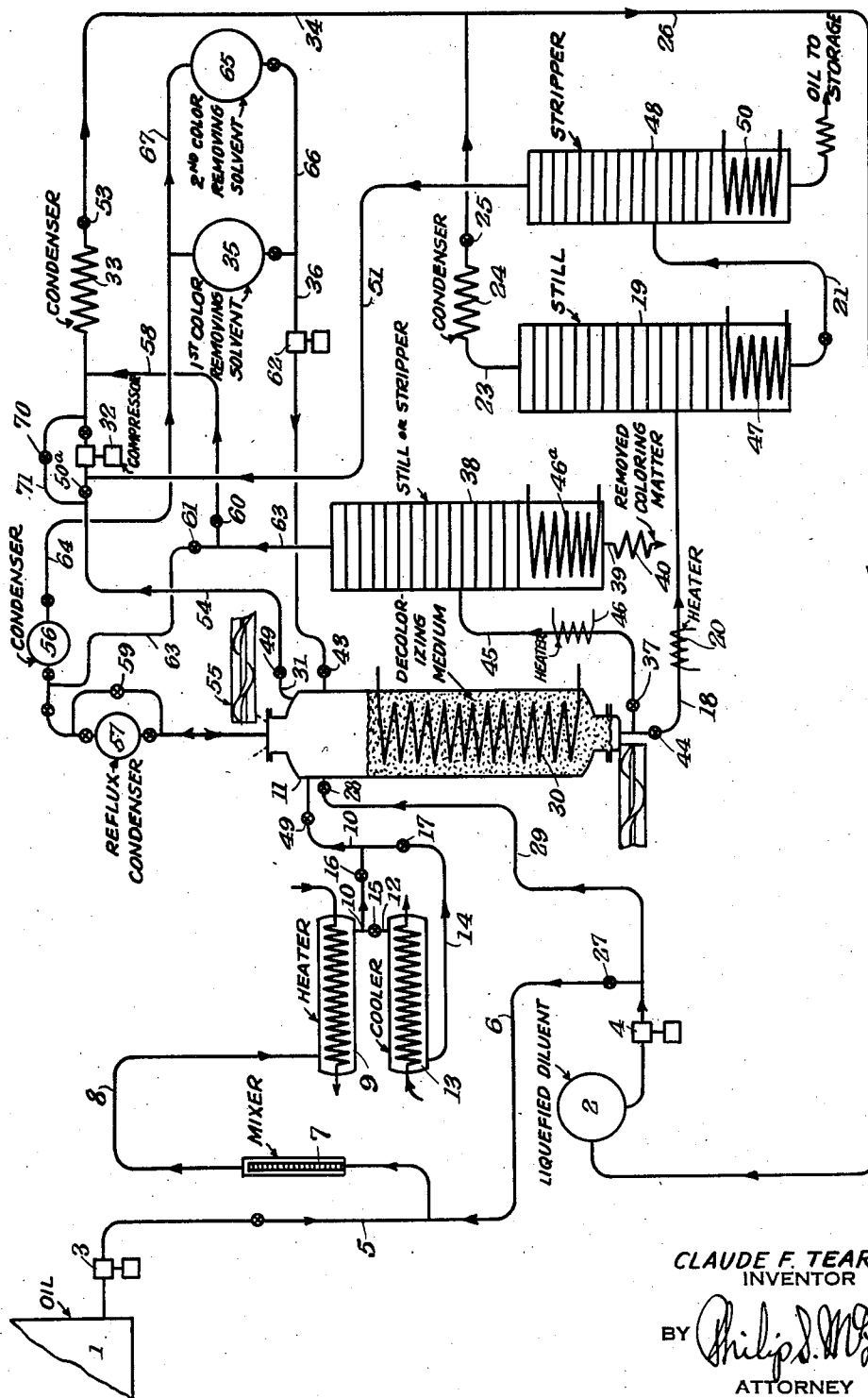
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REACTIVATION OF OIL DECOLORIZING MEDIUMS

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REACTIVATION OF OIL DECOLORIZING
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8 Claims. (Cl. 252—2)

This invention relates to the reactivation of the mediums employed for the decolorizing of lubricating oils and is a continuation in part of the Tears application, Serial No. 679,073, Patent 2,067,802, Jan. 12, 1937.

The conventional method of reactivating fuller's earth and like decolorizing mediums is by roasting. The expense incurred in the transfer to and from the roaster and the resultant losses are considerable.

Special objects of this invention are to avoid such expenses and losses and to get a greater efficiency in the reactivated material than has been possible by such prior methods.

These particular objects and other desirable objects are attained in the present invention by the novel features of invention hereinafter described, illustrated and broadly covered in the claims.

The drawing accompanying and forming part of the specification illustrates diagrammatically one preferred embodiment of the invention, but it will be understood that structure and steps of the process may be modified, as conditions require, all within the true intent and broad scope of the invention.

The invention involves reactivation of the decolorizing medium by color removing solvent. In the prior Tears application No. 679,073, above referred to, trichlorethylene was disclosed as being particularly effective in removing coloring matter, oil, or carbon from a propane treated clay and for reactivating such clay. It has been found that methylene chloride (dichlor-methane CH_2Cl_2) is especially effective as a color removing solvent and reactivating agent. Chloroform (CHCl_3) also has been used with success. These, as well as the general class, of the chlorinated hydrocarbon compounds, are peculiarly effective solvents for color-removal and reactivation.

To illustrate the process, reference is made to the accompanying diagram, which is in the nature of a flow sheet showing basically the percolation method of decolorizing first disclosed in the above-identified earlier Tears application and involving the filtering of lubricating oil in solution in a normally gaseous, liquefied hydrocarbon.

In this particular illustration, the oil to be decolorized is taken from storage tank 1 and the normally gaseous liquefied hydrocarbon, such as propane, under pressure, and in the liquid state, is taken from a suitable holder 2, pumps 3 and 4 conveying the same under pressure through lines 5 and 6 into a mixer 7. To promote complete

solution of oil in the solvent the mixture is shown as passing through line 8 to a heater 9. The heated solution may then be passed through line 10 direct to the filter 11 or it may first be passed by line 10—12 through cooler 13 and thence by line 14—10 to the filter, the cooling thus effected enabling operation at lower pressure. Valves 15, 16, 17 in the lines connecting the heater and cooler and the lines connecting with the filter enable such control to be exercised and, if desired, the sending of the flow partly through the heater and partly through the cooler in series with the heater.

It has been found that the most desirable filtering temperature is in the neighborhood of 125° F., but acceptable results can be obtained within the temperature range of 60° F. to 200° F.

Since the solvent is normally gaseous, it is necessary to maintain pressure on the entire mixing and filtering system sufficient to keep the solvent in the liquid state. This is effected in the illustration by a regulatable valve 49 in the flow line 10 leading to the filter, which maintains a desired back pressure on the mixing, heating and cooling system and valve 44 in the discharge line 18 from the bottom of the filter maintains a desired pressure in the filter.

The filtered or decolorized oil solution passes through line 18 to the still 19 through a heater 20 where it is heated to vaporize the solvent. The major portion of the solvent is distilled off in still 19 and leaving at the top by line 23 is condensed in condenser 24 and under control of valve 25 returned through line 26 to the storage tank 2.

Pressure of approximately 200 pounds per square inch is maintained on the still by control of valve 25 and heat for distillation is shown as applied by means of a heating coil 47 in the base of the still. From the bottom of the still the partially denuded, decolorized oil flows by line 21 to a stripper 48, which is provided with a heating coil 50 and is operated at low pressure, for instance on the order of 15 pounds per square inch. At this pressure all the remaining solvent may be distilled off through line 51, connected to the suction side of the compressor 32, which latter serves to compress the solvent vapor for recovery by condensation in the condenser 33. Back pressure is maintained in the condenser by manipulation of valve 53 and the recovered solvent in liquid form returns to the solvent storage tank 2 through lines 34—26. A valve 70 is shown provided in a bypass 71 about the compressor.

When the decolorizing clay becomes exhausted and therefore useless as a decolorizing medium, the oil is first washed out of the clay by pumping fresh solvent under pressure from pump 4 through line 29, with valve 28 open and valve 27 closed, directly into the top of the filter. This fresh solvent passes down through the clay and washes out the oil retained by the clay. This filter wash is passed over through line 18 to the still 19 for recovery of the solvent.

The next step is to remove the solvent from the clay for recovery. This may be accomplished by heating the bed of clay by means of the internal heater 30 and vaporizing the solvent through line 31 directly to the condenser 33 by opening the compressor bypass 71 at the valve 70. This distillation may be continued so long as solvent can be thus removed and at the end of such time the compressor bypass is closed at valve 70 and valve 50a is opened to vent the filter to the suction side of the compressor. In this operation all solvent is removed from the clay by low pressure distillation. After all the solvent has been thus removed, the clay is ready for reactivation by washing with the color-removing solvent.

The color-removing solvent is transferred by pump 62 from tank 35 through line 36 to the filter. Valve 44 at the bottom of the filter is closed and valve 37 in the line 45 leading to the still 38 is opened so that the solution of coloring matter in the color-removing solvent passes to still 38, being heated on the way by heater 46. Additional heat is supplied in the illustration by heater 46a in the base of the still. As the color-removing solvent is distilled off, the color-bearing constituents are removed at the bottom through line 39 and cooler 40. Such material may be diverted to a storage tank, not shown.

The overhead vapor from still 38 may be directed through line 63 to condenser 56 to be condensed and returned through line 64 to tank 35, or it may be passed through the reflux condenser 57 to be condensed and returned hot to the filter for the removal of additional coloring matter. When the reflux condenser is used in this way it is preferable to have a portion of the vapor stream passing through the final condenser 56 and back to the color-removing solvent storage tank 35.

Washing with the color-removing solvent is continued until the stream from the base of the filter is colorless, indicating complete removal of the color-bearing constituents from the decolorizing medium. It then becomes necessary to recover the color-removing solvent. This may be accomplished by distilling it from the decolorizing medium directly to condenser 56 through the valved bypass 59 around the reflux condenser 57, heat for such distillation being supplied by heating coil in the filter.

Distillation of the color-removing solvent is continued until all such solvent is removed from the decolorizing medium. In some cases it may be desirable to remove the last traces of color-removing solvent from the decolorizing medium by washing such medium with fresh, normally gaseous liquefied hydrocarbon from tank 2. When this washing step is employed the liquefied hydrocarbon solvent is passed on into still 38 from which it is distilled overhead through line 63—58 directly to condenser 33 by opening valve 60 and closing valve 61. Experience has shown that only a relatively small amount of the color-removing solvent is washed out of the decoloriz-

ing medium by this normally gaseous, liquefied hydrocarbon wash. The small amount thus removed may be accumulated in the base of still 38 and removed by distillation through condenser 56 when the still 38 is next used for recovery of color-removing solvent.

Decolorizing medium, such as 30–60 mesh fuller's earth may be reactivated at least six times using methylene chloride in the manner described and the reactivated clay has a relative efficiency of above 80% as compared with new fuller's earth. This is considerably higher than the efficiency of fuller's earth reactivated under the present methods of roasting.

After about six reactivations under this invention the efficiency of the decolorizing medium decreases rather rapidly. Apparently such sudden drop in decolorizing efficiency is due to failure of the methylene chloride to completely remove all the color-bearing compounds retained by the decolorizing medium. To further use the medium it then becomes necessary to employ a second color-removing solvent which will remove those compounds not removed by the methylene chloride. A desirable second solvent for such purpose is a mixture of 50% benzol and 50% methanol.

After reactivating the decolorizing medium some five or six times with methylene chloride, it may be washed with this second, benzol-methanol mixture to reactivate it for use again. In the illustration, the benzol-methanol mixture is supplied from tank 65 and line 66—36, by pump 62 to the filter and returned after recovery to the tank by line 64—67.

The process has been described above in connection with percolation filtration of a solution of lubricating oil in normally gaseous liquefied hydrocarbon. While the use of normally gaseous liquefied hydrocarbon solvent is highly desirable in the operation of the present process, it is not entirely essential and the reactivation need not necessarily involve the washing with normally gaseous liquefied hydrocarbon solvent before and after washing with the color-removing solvent. Other oil solvents may be used for removal of oil from the fuller's earth and for the removal of the color-removing solvent from the fuller's earth.

The present solvent reactivating process may be employed also for reactivation of contact filtering mediums, such as fine fuller's earth, or some of the chemically treated decolorizing clays. Such contact filtering operations may be conducted with the oil in solution in a normally gaseous liquefied hydrocarbon solvent, in naphtha as a solvent, or the oil may be contacted hot without the use of any solvent. In all such contacting operations it is usual to filter out the contact clay in a filter press, the clay being retained on the filter plates. A convenient means of reactivating such contact clay under the present invention is to wash it in place on the filter plates with the color-removing solvents here disclosed. The clay can then be dried in place in the filter, or may be removed from the filter and dried elsewhere, being then ready for use again.

As an example of the effectiveness of the present method, ordinary, fine fuller's earth was used for contact filtering a solution of Pennsylvania steam-refined stock in naphtha at a temperature of 150° F. with three pounds of the fine fuller's earth per gallon of steam-refined stock in the solution. This resulted in a color of 8 N. P. A. after the naphtha was removed from the de-

colorized oil. The contact clay was reactivated in situ on the filter while washing with chloroform until the color of the chloroform indicated removal of no more color constituents. This same
5 clay was used five times after such reactivation and under the same filtering conditions it was found that there was no distinguishable differences in the color of the finished oil from the five filtrations.

10 The present invention avoids the costly handling and transfer between the filter and roaster and saves the losses heretofore occasioned by such handling. Furthermore, the efficiency of the medium reactivated by this solvent washing is
15 much higher than the same medium reactivated by the roasting. The invention may be practiced by relatively simple additions to and modification of present equipment.

I claim:

20 1. The herein disclosed process of reactivating adsorbent decolorizing medium, which comprises washing the same with methylene chloride.

25 2. The herein disclosed process of reactivating adsorbent decolorizing medium, which comprises washing the same with methylene chloride and thereafter washing said medium with a mixture of benzol and methanol.

30 3. The herein disclosed method of reactivating adsorbent decolorizing medium which comprises first washing the same with normally gaseous liquefied hydrocarbon and then washing said medium with methylene chloride.

35 4. The process of reactivating exhausted adsorbent decolorizing medium, which comprises washing the same with a color-removing solvent of the chlorinated hydrocarbon class and then washing the medium with normally gaseous liquefied hydrocarbon while maintaining sufficient

pressure to hold the normally gaseous liquefied hydrocarbon in a liquid state.

5 5. The process of reactivating decolorizing medium, which comprises washing the same with a color-removing solvent of the chlorinated hydrocarbon class, then washing with a mixture of benzol and methanol and finally washing with a normally gaseous liquefied hydrocarbon under pressure sufficient to maintain the solvent in the liquid state. 10

6. The process of reactivating decolorizing medium, which comprises washing the same with methylene chloride, washing with a benzol-methanol mixture, and then washing with a liquefied normally gaseous hydrocarbon under pressure
15 sufficient to maintain the solvent in the liquid state to remove traces of both previous washes.

7. The process of repeatedly reactivating adsorbent decolorizing medium, which comprises
20 washing said decolorizing medium after each use with chlorinated hydrocarbon solvent until such washing fails to restore the decolorizing efficiency of the adsorbent medium and then washing said medium with a mixture of methanol and benzol, which will remove the color compounds irremov-
25 able by the chlorinated hydrocarbon and thereafter washing with a liquefied normally gaseous hydrocarbon under sufficient pressure to maintain the same in a liquid state.

8. The process of reactivating decolorizing
30 medium which comprises washing it first with a liquefied normally gaseous hydrocarbon under pressure sufficient to maintain the same in a liquid state, then washing with a chlorinated hydrocarbon and thereafter washing with a liquefied
35 normally gaseous hydrocarbon under pressure sufficient to maintain the same in a liquid state.

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