Patented Feb. 24, 1942 2,273,846

UNITED STATES PATENT OFFICE

2,273,846

PROCESS FOR TREATING HYDROCARBONS

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No Drawing. Application August 25, 1941, Serial No. 408,260

14 Claims. (Cl. 196—16)

My invention relates generally to the treatment of hydrocarbons and has for one of its objects the refining or the re-refining of such materials.

The present patent application is a continuation in part of my copending patent application, Serial No. 241,960, filed November 23, 1938.

More specifically my invention relates to the treatment of oils such as mineral oils of a lubricating nature or of an insulating nature, and mineral waxes, etc., all of which are subject to more or less deterioration when exposed to air, moisture, metals or other agents which cause contamination such as oxidation, polymerization, etc. as well as contamination by solid materials in suspension.

In the case of lubricating oils, for example, it has been found that although the most modern methods are used in the refining of these oils for lubricating purposes yet certain undesirable properties remain in such lubricants which should be removed. It is known also that in use, lubricating oils are subject to contamination by water, free and in solution, metallic particles, dust and other solids and air both free and dissolved. It has been discovered, also, that varying shock loads detrimentally affect such oils. Under such trying conditions many materials are produced in the oil which deleteriously affect the same so far as its use as a lubricant is concerned.

Without attempting to exhaust the subject, I might say that these contaminants produce sludges due to excessive heating, free and amorphous carbon, stable and unstable emulsions, polymerization products, soluble and insoluble resins and gums, acids, metallic soap, asphaltic sludges; free acids, dissolved gases and free water as well as addition agents are present in the oil. The oil also becomes discolored. So far as a lubricating oil is concerned, the presence of these various materials in the oil causes excessive bearing wear, over-heated bearings, increases internal oil friction, reduces the film strength, etc.

Insulating oils are subjected to contamination and deterioration as the result of contact of the heated hydrocarbon with air or certain other gases, both free and in solution, and contact of the oil with metal surfaces. For example, in the case of insulating oils used with circuit breakers I have found that the presence of air which contains water vapor, oxygen, nitrogen, dust, etc., causes slow sludging and water condensation. The arc action of the circuit breaker produces carbon in suspension and precipitated; carbon is also precipitated on the terminals and oil gases and hydrogen are produced as well as some unsaturates. The effect of this is rapid oil deterioration from the arc, carbonization of the oil, decreased rupture capacity, explosion hazard and possible failure of the breaker. In the case of electric transformers, water, gas in solution, organic fluids and volatiles, organic acids, sludges, resinous bodies and compounds, tars, pitches and oxyasphaltic compounds are produced due to the heat from operation of the transformer. This, of course, lowers the insulation value of the oil, prevents proper heat dissipation, induces gas pocket formation, creates explosion hazards and volatile corrosion, promotes power factor losses and possible failure of the equipment. I might say that in the case of oil-insulated electric cables the high electric stresses due to operation of the cable causes deterioration of the insulating oil in much the same fashion as in the case of transformers, the effect of which may be failure of the cable due to ionization, wax formation, a decrease in resistivity and an increase in the power factor.

In the case of mineral waxes I have found that the products of oxidation together with free water and dissolved gases produce discoloration of the mineral wax and in many cases liberates unsaturated compounds and forms insoluble resins, gums, soaps and various types of sludge impurities. These vary slightly in degree with the type of mineral wax.

The present invention provides a process for the removal of the impurities or contaminants above referred to.

My process, it is to be noted, may be continuous or intermittent.

Broadly speaking, my improved process is directed to the treatment of the oils and waxes above referred to wherein I agitate a mixture of the oil or wax to be treated and an adsorbent material while the mixture is at an elevated temperature and under vacuum. After this treatment the mixture is filtered.

More specifically, in practicing my invention the material to be treated is introduced into a heating and mixing chamber which is hermetically sealed and connected to any suitable commercial type fractional condenser. A suitable adsorbent material, such as bone char, carbon black, fuller's earth, etc., is added to the charging stock in the heating and mixing chamber. The amount of adsorbent material employed so far as maximum amount is concerned is not

critical except perhaps for economical reasons. For example, I may add from one-half per cent to twelve per cent, or even as high as forty per cent in special cases, by weight of the oil or wax being treated or processed. In addition to the adsorbent material I prefer to add water and in some cases superheated steam as a flushing agent and an alkali such as potassium hydroxide or calcium hydroxide or the like to bring about saponification of any residual acid which is present and which is probably due to oxidation of the oil or wax due to contact of the heated material with air. I have found that this residual acid is not readily adsorbable by the adsorbent material of the mixture. The addition of the potassium hydroxide saponifies this acid as above mentioned so that the same may be readily removed.

The amount of water and alkali added may be varied within a wide range. For example, I may say that the amount of water or steam added may vary between one-quarter of a pound and one pound per gallon of material being treated and the amount of alkali may vary from 0.02% to 1% by weight.

The heating and mixing chamber is hermetically sealed, as above noted, and is preferably under vacuum, to obtain perfect or superior re-refined products although good results are obtained if the vacuum is not employed. The mixture in the mixing chamber is agitated rapidly by any suitable commercial agitator such as a motor driven pump or a turbine impellor while the mixture is being raised to an elevated temperature in a range sufficient to drive off the water and the low boiling point volatile impurities. The temperature range extends up to a value in the neighborhood of the flash point of the oil or wax in its finally treated state and includes an elevated temperature below the flash point of the finally treated oil or wax and preferably embraces a range of approximately 50 to 150 degrees F. below the flash point of the finally treated oil or wax. It will be appreciated, of course, that at such a temperature the water, light ends and volatile oxidation products will be driven off and flow into the condenser which, as above noted, is connected to the mixing and heating chamber. Because of thermal decomposition or deterioration, the heating of the mixture at a temperature substantially beyond the flash point of the finally treated oil or wax causes a material reduction in the yield of the oil or wax being treated. At temperatures below the flash point of the finally treated oil or wax, the yield is good, but as the temperature substantially exceeds the flash point of the finally treated oil or wax, the yield rapidly decreases, rendering the process uneconomical.

The mixture is maintained at this elevated temperature and subjected to agitation for from fifteen minutes to an hour, for example.

As above mentioned, I prefer to employ a vacuum on the heating and mixing chamber, although good results are obtained without the employment of a vacuum. If a vacuum is employed I prefer a high vacuum from twenty-eight to thirty inches of mercury as referred to a thirty inch barometer at sea level. The purpose of utilizing a vacuum is to increase the rate of distillation at a lower temperature of oils which are quite sensitive to the action of heat, especially those of a low viscosity.

The above remarks apply also to the treatment of mineral waxes.

Hydrogenization of the liquids being treated may also be accomplished during treatment and while in the presence of adsorbent materials which may or may not have some definite action as catalysts.

For example, in the treatment of certain mineral waxes I am able to produce a pure white wax which may be used in the manufacture of artificial honeycombs from which all traces of odor are removed. In the case of electrical insulating oils the high vacuum accelerates the removal of the free water of solution as well as all gases with which the oil may be saturated and which would deleteriously affect the electrical and physical characteristics of this class of oil.

After this treatment of the material in the heating and mixing chamber I pass the material through a filter in a closed filter chamber. Various forms of filter materials may be employed. For example, paper, glass cloth, glass wool or metallic screens, any or all of which may be protected with a diatomaceous filter aid, so as to secure the maximum degree of clarity of the material being treated. The material may be pumped through the filter or drawn through.

It will be understood that in the filtering operation the adsorbent material with it adsorbed impurities such as resins, metallic soaps, heavy residual acids, polymer products, mechanical impurities, etc., are removed.

The treated material is now in a clean condition suitable for use or service.

It will be seen from all of the foregoing that the present invention provides a process for the treatment of oils and waxes, whereby contaminants in these materials which deleteriously affect the same are removed, the process being adapted for use in connection with the refining of such oils and waxes as well as to the re-refining of the same after they have been in service.

The specification and the claims are directed to a process for removing impurities from a contaminated or used mineral substance of the class consisting of mineral oils and mineral waxes. The term "contaminated" as used herein means an oil or wax which is unfit for a specific purpose.

It is to be understood, of course, that changes may be made in the procedures above described without departing from the spirit and scope of my invention.

I claim as my invention:

1. The process for removing impurities from a contaminated or used mineral substance of the class consisting of mineral oils and mineral waxes, which process comprises mechanically mixing and agitating the contaminated or used mineral substance and a solid adsorbent material in a sealed container to adsorb the high boiling point impurities, heating the mixture of the mineral substance being treated and the solid adsorbent material in the sealed container under vacuum at a temperature in the range of approximately 50 to 150 degrees F. below the flash point of the finally treated mineral substance to drive off the water and the low boiling point volatile impurities, and separating the adsorbent material with its adsorbent impurities from the mineral substance being treated to recover the latter.

2. The process for removing impurities from a contaminated or used mineral oil, which process comprises mechanically mixing and agitating the contaminated or used oil and a solid adsorbent material in a sealed container to adsorb the high boiling point impurities, heating the mixture of the oil being treated and the solid adsorbent material in the sealed container under vacuum at a

temperature in the range of approximately 50 to 150 degrees F. below the flash point of the finally treated oil to drive off the water and the low boiling point volatile impurities, and separating the adsorbent material with its adsorbed impurities from the oil being treated to recover the latter.

3. The process for removing impurities from a contaminated or used mineral wax, which process comprises mechanically mixing and agitating the contaminated or used wax and a solid adsorbent material in a sealed container to adsorb the high boiling point impurities, heating the mixture of the wax being treated and the solid adsorbent material in the sealed container under vacuum at a temperature in the range of approximately 50 to 150 degrees F. below the flash point of the finally treated wax to drive off the water and the low boiling point volatile impurities, and separating the adsorbent material with its adsorbed impurities from the wax being treated to recover the latter.

4. The process for removing impurities from a contaminated or used mineral oil, which process comprises mechanically mixing and agitating the contaminated or used oil, a solid adsorbent material, an alkali, and an aqueous medium of the class consisting of water and steam in a sealed container to adsorb the high boiling point impurities, heating the mixture of the oil being treated, the adsorbent material, the alkali and the aqueous medium in the sealed container under vacuum at a temperature in the range of approximately 50 to 150 degrees F. below the flash point of the finally treated oil to drive off the water and the low boiling point volatile impurities, and separating the adsorbent material with its adsorbed impurities from the oil being treated to recover the latter.

5. The process for removing impurities from a contaminated or used mineral substance of the class consisting of mineral oils and mineral waxes, which process comprises mechanically mixing and agitating the contaminated or used mineral substance, a solid adsorbent material and an aqueous medium of the class consisting of water and steam, in a sealed container to adsorb the high boiling point impurities, heating the mixture of the mineral substance being treated, the solid adsorbent material and the aqueous medium in the sealed container under vacuum at a temperature in the range of approximately 50 to 150 degrees F. below the flash point of the finally treated mineral substance to drive off the water and the low boiling point volatile impurities, and separating the adsorbent material with its adsorbed impurities from the mineral substance being treated to recover the latter.

6. The process for removing impurities from a contaminated or used mineral substance of the class consisting of mineral oils and mineral waxes, which process comprises mechanically mixing and agitating the contaminated or used mineral substance and a solid adsorbent material in a sealed container to adsorb the high boiling point impurities, heating the mixture of the mineral substance being treated and the solid adsorbent material in the sealed container under vacuum at a temperature below the flash point of the finally treated mineral substance to drive off the water and the low boiling point volatile impurities, and separating the adsorbent material with its adsorbed impurities from the mineral substance being treated to recover the latter.

7. The process for removing impurities from a contaminated or used mineral oil, which process comprises mechanically mixing and agitating the contaminated or used oil and a solid adsorbent material in a sealed container to adsorb the high boiling point impurities, heating the mixture of the oil being treated and the solid adsorbent material in the sealed container under vacuum at a temperature below the flash point of the finally treated oil to drive off the water and the low boiling point volatile impurities, and separating the adsorbent material with its adsorbed impurities from the oil being treated to recover the latter.

8. The process for removing impurities from a contaminated or used mineral wax, which process comprises mechanically mixing and agitating the contaminated or used wax and a solid adsorbent material in a sealed container to adsorb the high boiling point impurities, heating the mixture of the wax being treated and the solid adsorbent material in the sealed container under vacuum at a temperature below the flash point of the finally treated wax to drive off the water and the low boiling point volatile impurities, and separating the adsorbent material with its adsorbed impurities from the wax being treated to recover the latter.

9. The process for removing impurities from a contaminated or used mineral oil, which process comprises mechanically mixing and agitating the contaminated or used oil, a solid adsorbent material, an alkali, and an aqueous medium of the class consisting of water and steam in a sealed container to adsorb the high boiling point impurities, heating the mixture of the oil being treated, the adsorbent material, the alkali and the aqueous medium in the sealed container under vacuum at a temperature below the flash point of the finally treated oil to drive off the water and the low boiling point volatile impurities, and separating the adsorbent material with its adsorbent impurities from the oil being treated to recover the latter.

10. The process for removing impurities from a contaminated or used mineral substance of the class consisting of mineral oils and mineral waxes, which process comprises mechanically mixing and agitating the contaminated or used mineral substance, a solid adsorbent material and an aqueous medium of the class consisting of water and steam, in a sealed container to adsorb the high boiling point impurities, heating the mixture of the mineral substance being treated, the solid adsorbent material and the aqueous medium in the sealed container under vacuum at a temperature below the flash point of the finally treated mineral substance to drive off the water and the low boiling point volatile impurities, and separating the adsorbent material with its adsorbed impurities from the mineral substance being treated to recover the latter.

11. The process for removing impurities from a contaminated or used mineral substance of the class consisting of mineral oils and mineral waxes, which process comprises mechanically mixing and agitating the contaminated or used mineral substance, a solid adsorbent material, an alkali, and an aqueous medium of the class consisting of water and steam in a sealed container to adsorb the high boiling point impurities, heating the mixture of the mineral substance being treated, the adsorbent material, the alkali and the aqueous medium in the sealed container under vacuum at a temperature in the range of 50 to 150 degrees F. below the flash point of the finely treated oil to drive off the water and the low

boiling point volatile impurities, and separating the adsorbent material with its adsorbed impurities from the mineral substance being treated to recover the latter.

12. The process for removing impurities from a contaminated or used mineral substance of the class consisting of mineral oils and mineral waxes, which process comprises mechanically mixing and agitating the contaminated or used mineral substance, a solid adsorbent material, an alkali, and an aqueous medium of the class consisting of water and steam in a sealed container to adsorb the high boiling point impurities, heating the mixture of the mineral substance being treated, the adsorbent material, the alkali and the aqueous medium in the sealed container under vacuum at a temperature below the flash point of the finely treated oil to drive off the water and the low boiling point volatile impurities, and separating the adsorbent material with its adsorbed impurities from the mineral substance being treated to recover the latter.

13. The process for removing impurities from a contaminated or used mineral wax, which process comprises mechanically mixing and agitating the contaminated or used wax, a solid adsorbent material, an alkali, and an aqueous medium of the class consisting of water and steam in a sealed container to adsorb the high boiling point impurities, heating the mixture of the wax being treated, the adsorbent material, the alkali and the aqueous medium in the sealed container under vacuum at a temperature in the range of approximately 50 to 150 degrees F. below the flash point of the finally treated wax to drive off the water and the low boiling point volatile impurities, and separating the adsorbent material with its adsorbed impurities from the wax being treated to recover the latter.

14. The process for removing impurities from a contaminated or used mineral wax, which process comprises mechanically mixing and agitating the contaminated or used wax, a solid adsorbent material, an alkali, and an aqueous medium of the class consisting of water and steam in a sealed container to adsorb the high boiling point impurities, heating the mixture of the wax being treated, the adsorbent material, the alkali and the aqueous medium in the sealed container under vacuum at a temperature below the flash point of the finally treated wax to drive off the water and the low boiling point volatile impurities, and separating the adsorbent material with its adsorbed impurities from the wax being treated to recover the latter.

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