

UNITED STATES PATENT OFFICE

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PROCESS OF PURIFYING LUBRICATING-OIL DISTILLATES

No Drawing.

Application filed July 31, 1928. Serial No. 296,629.

This invention relates to a process of purifying viscous mineral oil fractions for the production of lubricating oils, to extract therefrom constituents which operate to produce emulsions when commingled with water, or a water solution of caustic soda, and which also operate to hinder decolorization of the oil.

An object of the invention is to purify mineral lubricating oil distillates to produce a treated oil which will not form persistent emulsions with water, a water solution of caustic soda or water containing dissolved mineral salts, such as sea water.

Another object of the invention is to provide a process of treating viscous petroleum oil distillates without the formation of persistent emulsions during treatment thereof and without any substantial loss of the finished lubricating oil.

Another object of the invention is to produce a lubricating oil suitable for lubricating steam turbines or other machinery in which the lubricant may come into contact or become commingled with water or steam.

Another object of the invention is to produce a lubricating oil free from emulsifying constituents such as resins, gums, organic acids, phenols, or organic sulpho-acids.

Other objects and advantages will appear in the practical use of the same from the following description.

During the treatment of lubricating oil stocks derived from an asphalt base crude petroleum oil, with sulphuric acid, certain emulsifying agents are formed, such as organic acids, oxy and oxy-sulpho-acids, which have a preferential oil solubility and remain in solution in the treated lubricating oil stock after the acid sludge has been separated from the oil. These emulsifying constituents cannot be completely removed from the oil by any of the well-known methods of neutralizing with caustic soda and washing with water.

By these well-known methods of neutralizing an acid treated lubricating oil with caustic soda and washing with water, a certain percentage of the treated lubricating oil is emulsified with the water solution of caus-

tic soda and the removal thereof diminishes the yield of said treated oil. Also, a portion of the said emulsifying constituents remain in the treated oil, which causes the same to form emulsion when used to lubricate steam turbines in which the oil comes into contact with water or sea-water employed in the cooling system. In particular, in the manufacture of lubricating oils from an asphalt or mixed base crude petroleum oil, by well-known methods, there is lost during the neutralizing and washing operation from 5 to 15 per cent by volume of the treated oil, and under the most favorable conditions only an imperfect separation of the emulsifying constituents is obtained.

Certain methods have been heretofore developed with some success to employ alcohol and other like solvents to remove the emulsifying constituents which occur naturally or those produced in the acid treatment of lubricating oil stocks.

I have discovered that superior results can be obtained by performing the neutralization operation in two steps, the first step comprising adding and commingling with the acid treated lubricating oil, a water solution of caustic soda in quantities just sufficient to substantially neutralize all the sulphuric and sulphurous acid present, and without any substantial neutralization of the organic or sulpho-organic acid constituents present, whereby oil and water emulsions are not formed. The quantity of the water solution of caustic soda required to neutralize the sulphuric and sulphurous acids present is determined by titrating a sample of the acid-treated lubricating oil stock with the water solution of caustic soda to be employed, using any of the well known indicators which are not sensitive to organic acids, such as methyl orange. After this first neutralizing step, the lubricating oil stock is separated from the water containing the products of neutralization and is then completely neutralized by commingling with a water solution of potassium hydroxide and steam at a pressure of approximately 50 pounds gauge. The introduction of steam is continued until a temperature of approximately 250 to 280 de-

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degrees F. is attained, after which the introduction of steam is discontinued and the water solution of salts and excess potassium hydroxide separated therefrom.

5 By this method a complete neutralization and separation of substantially all the emulsifying constituents is effected and the treated lubricating oil stock is thereafter decolorized to the required degree by methods known in the art.

10 The following describes the preferred method of carrying out this invention.

For illustration, a lubricating oil distillate of 18 to 21 degrees Bé. derived by vacuum distillation of an asphalt or mixed base crude petroleum oil, may first be treated with 15 1/4 pound of 66 degree Bé. sulphuric acid per gallon of oil and subsequently with successive applications of from 1/4 to as high as 2 pounds or higher of sulphuric acid having a strength 20 ranging from 93 per cent sulphuric acid to as high as 15 per cent fuming sulphuric acid per gallon of oil, this treatment depending upon the stock treated and the product desired, the acid being agitated with the oil 25 and the sludge formed being removed prior to the application of each succeeding portion of acid used.

After the removal of substantially all of 30 the acid sludge from the treated oil, it is in condition to be neutralized and freed from the emulsifying constituents, which is carried out by preferably transferring the treated oil to a second agitator or treating 35 tank provided with heating means such as a steam coil. The acid treated oil in this second agitator is then commingled with a water solution of caustic soda added in quantities just sufficient to neutralize the free sulphur 40 dioxide or sulphurous acid and free sulphuric acid present, and without any substantial neutralization of the organic or sulpho organic acid constituents present, with the application of heat at a temperature of approximately 150 to 180 degrees F., after which the 45 water containing the products of neutralization free from oil emulsions, is separated from the partly neutralized treated oil by withdrawing the same from the bottom of 50 the agitator or treating tank after settling.

After this first partial neutralizing step and separation of the oil from the water and products of neutralization, the oil is commingled with a water solution of potassium 55 hydroxide in quantities sufficient to completely neutralize all the organic and sulpho acid constituents present, preferably in the same agitator and then transferring the same in a commingled state to a pressure treating 60 tank provided with means for introducing steam into the commingled mixture under pressure. Steam is then introduced into the neutralized treated oil mixture until a temperature of approximately 250 to 280 degrees 65 F. is obtained at a pressure of approximately

50 pounds gauge, after which the steam is shut off and the treated oil permitted to separate from the water component which contains the emulsifying constituents in solution as water soluble potassium salts, together with the excess potassium hydroxide employed in the neutralization operation, which is separated from the oil by withdrawing from the bottom of the said pressure treating tank. The purified lubricating oil stock now free of emulsifying constituents, is preferably again distilled under a vacuum in order to separate the various grades of lubricating oil stocks and thereafter clarified and decolorized to the required degree by the employment of a decolorizing agent, or the treated lubricating oil stock may be clarified and decolorized to the required degree by methods known in the art, without a re-distillation operation, or may be employed directly without further treatment.

By the term "free mineral acids" I mean uncombined sulphur dioxide, sulphurous, or sulphuric acid, which may be present in the lubricating oil stock after treatment with sulphuric acid.

While the process herein described is well adapted for carrying out the object of the invention, it is to be understood that various changes and modifications may be made without departing from the spirit of the invention and the invention includes all such changes and modifications as come within the scope of the appended claims.

What I claim is:

1. That step in the process of purifying mineral lubricating oil stocks after treatment with sulphuric acid and removal of the acid sludge therefrom, comprising, first neutralizing and separating the free mineral acids as water soluble mineral acid salts, from the acid treated lubricating oil stock, without any substantial neutralization of the organic acid, or sulpho-acids present, then neutralizing the organic acids and sulpho-acids by commingling the acid treated oil from which the said mineral acids have been removed, with a water solution of potassium hydroxide and finally separating the neutralized organic acids and sulpho organic acids from the treated oil by subjecting the treated oil commingled with the water solution of potassium hydroxide to a temperature of approximately 250 to 280 degrees F. at a pressure of approximately 50 pounds gauge, produced by the introduction of steam into the mixture.

2. That step in the process of purifying mineral lubricating oil stocks after treatment with sulphuric acid and removal of the acid sludge therefrom, comprising, first neutralizing the free mineral acids in the acid treated lubricating oil stock with a water solution of caustic soda introduced in quantities just sufficient to substantially neutralize all free

mineral acids present, without any substantial neutralization of the organic acid emulsifying constituents present, separating and removing the water solution of mineral acid salts from the lubricating oil stock and then commingling the lubricating oil stock from which the free mineral acids have been removed, with a water solution of potassium hydroxide in quantities sufficient to neutralize the organic acid emulsifying constituents present and then separating a water solution of the neutralized organic acid emulsifying constituents from the lubricating oil stock by subjecting the same to a temperature of approximately 250 to 280 degrees F. at a pressure of approximately 50 pounds gauge.

3. That step in the process of purifying mineral lubricating oil stocks after treatment with sulphuric acid and removal of the acid sludge therefrom, comprising, neutralizing the free mineral acids in an acid treated lubricating oil stock with a water solution of caustic soda introduced in quantities just sufficient to substantially neutralize all free mineral acids present and without any substantial neutralization of the organic acid emulsifying constituents contained therein, separating and removing the water solution of mineral acid salts from the lubricating oil stock and then commingling the lubricating oil stock from which the free mineral acids have been removed, with a water solution of potassium hydroxide in quantities sufficient to neutralize all the organic acid emulsifying constituents present, and then separating a water solution of the neutralized organic acid emulsifying constituents from the lubricating oil stock by subjecting the same to a temperature of approximately 250 to 280 degrees F. at a pressure of approximately 50 pounds produced by the introduction of steam into the commingled mixture.

4. A process of purifying mineral lubricating oil stocks after treatment with sulphuric acid for the removal of emulsifying constituents, comprising, neutralizing and removing the free mineral acids from an acid treated lubricating oil stock from which the acid sludge has been substantially removed, with a water solution of caustic soda, without any substantial neutralization or removal of the organic acid constituents contained therein and then removing the organic acid constituents by commingling the said acid treated lubricating oil stock from which the free mineral acids have been removed, with an excess of a water solution of potassium hydroxide at a temperature of approximately 250 to 280° F., and finally separating the neutralized organic acids from the lubricating oil stock.

5. A process of purifying mineral lubricating oil stocks after treatment with sulphuric acid, for the removal of the emulsifying constituents, comprising, neutralizing

and removing the free mineral acids from an acid treated lubricating oil stock from which the acid sludge has been substantially removed, with a water solution of caustic soda without any substantial neutralization of the organic acid constituents contained therein and then removing the organic acid constituents by commingling the acid treated lubricating oil stock from which the free mineral acids have been removed, with an excess of a water solution of potassium hydroxide and finally separating a water solution containing the neutralized organic acid constituents from the lubricating oil stock at a temperature of approximately 250 to 280 degrees F. and at a pressure of approximately 50 pounds.

6. A process of purifying mineral lubricating oil stocks after treatment with sulphuric acid and removal of the acid sludge therefrom, comprising, neutralizing the free mineral acids in an acid treated lubricating oil stock, by commingling the same with the requisite quantity of a water solution of sodium hydroxide at a temperature of approximately 150 degrees F. without any substantial neutralization of the organic acid emulsifying constituents contained therein, separating and removing the water solution of mineral acid salts from the lubricating oil stock, and then commingling the lubricating oil stock from which the free mineral acids have been removed, with a water solution of potassium hydroxide in quantities sufficient to neutralize all the organic acid emulsifying constituents present, and then separating in a water solution containing the neutralized organic acid emulsifying constituents, from the lubricating oil stock by subjecting the commingled mixture of lubricating oil stock and potassium hydroxide to a temperature of approximately 250 to 280 degrees F. at a pressure of approximately 50 pounds.

In testimony whereof I affix my signature.
MARVIN L. CHAPPELL.

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