

UNITED STATES PATENT OFFICE.

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PROCESS OF MANUFACTURING A NONEMULSIFIABLE MINERAL OIL OF HIGH-DIELECTRIC STRENGTH.

No Drawing.

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The object of my invention is to manufacture refined mineral oil, and particularly lubricating oil, that will be non-emulsifiable and have a sufficiently high dielectric strength to adapt it for use with high voltage switches, transformers and turbines.

The invention involves a preliminary treatment of the oil for the purpose of removing therefrom the fatty acids contained therein as well as a substantial modification of the usual treatment for manufacturing refined mineral oil and particularly refined lubricating oil.

While the application of the process is not confined to any particularly kind of crude petroleum, the specific process would be necessarily varied somewhat with crude petroleum of different kinds, as, for example, those having an asphaltic base and those having a paraffin base. The specific process that I have used, and prefer to use, and which is herein described as the preferred embodiment of my invention, is one especially applicable to Texas oil and is believed to be equally applicable to all or most naphthenic oils; and should therefore not be understood as a necessarily preferable embodiment of the invention as applied to other kinds of crude oil, especially those with a paraffin base.

I have also herein described certain deviations from the preferred specific procedure which I have found permissible, although it will be understood that it would be impracticable to attempt to recite all possible deviations even with Texas oil, and much less with crude oil of a different character.

I first proceed to distill the crude petroleum, the distillation being preferably continued until only a substantially dry residue is left in the still. It is this distillate which I treat in the practice of the present process. With certain kinds of crude petroleum, it may be advisable to stop the distillation when certain heavy fractions are left in the still; or it may be advisable to distill off the fractions, remove the lighter fractions during the process of distillation, and treat, in accordance with my process, only the heavier fractions. The latter procedure may be advisable where the crude oil is rich in gasoline and it is desired to render non-emulsifiable and of high dielectric strength

only the lubricating oil, in which case the gasoline, naphtha, kerosene and gas oil will first be distilled off and the gas oil cracked to yield further quantities of gasoline, and my treatment applied only to the lubricating distillates.

Twenty-three hundred barrels of the distillate are run into an agitator and brought to a temperature most convenient for treatment. This temperature is a variable one, and is determined by the particular distillate used. With the naphthene distillate to which I preferably apply my process, the permissible range of temperature is between 80° F. and 160° F.

While the distillate is being agitated, I add an alkali, such as caustic soda, calcium hydrate, potassium hydrate, sodium carbonate, etc. The strength and amount of the alkali solution is controlled by the kind of distillate used. With the naphthene distillate to which I have applied my process, a strength of from 2 to 12 Baumé and quantity up to eighteen per cent of caustic soda have been found to give good results. As soon as the distillate shows a permanent alkaline reaction, the introduction of the alkali is stopped and the agitation is continued for an hour or so. This insures the thorough conversion of the fatty acids into soaps. The agitation is then discontinued and the distillate permitted to become quiet, allowing the soaps and excess alkali to settle. When the supernatant distillate is almost free from soaps, the soaps are drawn off.

The fatty acids, or, as they are otherwise sometimes termed, organic acids or saponifiable oils, are valuable by-products of my process and, for certain purposes, may be used in the form of soaps and in the condition in which they are drawn off as described, while for certain other purposes they are further purified. For the purposes of my invention, however, I have ascertained that the presence of these fatty acid soaps in the finished oil renders the oil emulsifiable and gives the oil a low dielectric strength, due, in part at least, to the water which is a necessary constituent of the soaps. I have also discovered that when these fatty acid soaps are removed from the distillate at the stage, and in the manner, specified, the distillate may be so

treated, with some modification of the usual process, as to produce a non-emulsifiable oil of high dielectric strength.

The distillate, freed of its fatty acids as above described, is then washed free of any suspended matter. This washing is with live steam. However, it may be washed first with water and then with steam, or washing with water and steam may proceed concurrently. However, if washed with water, the water washing must not follow the live steam wash, the latter being advisably, if not indeed necessarily, the last operation. The three methods of washing are equivalent in my process.

At this stage of the process the distillate may be subjected to a certain treatment and then reduced to stock or it may be reduced to stock and then subjected to a certain treatment.

Assuming that the distillate is treated before reduction: it is first brought to a convenient temperature, say 60-75° F., and intimately mixed with from two to six per cent by volume of sulfuric acid of preferably about ninety-eight per cent strength, although other strengths may be used with good results. It may be possible to introduce the acid in the more usual way, but in the practice of the process I have accomplished the mixing by agitation with air. Preferably I create a spray of compressed air and sulfuric acid in a finely divided state approximating a mist or vapor and forcibly direct the spray into the oil (as explained with more particularity in my Patent No. 1,272,979, dated July 16, 1918) while agitating the oil. The more intimate distribution of the acid insures greater contact and a more thoroughly purified oil, with a minimum quantity of acid and with less loss to the distillate. The oil is agitated about thirty minutes after the introduction of the acid and allowed to settle. After a period of time, most of the acid and sludge will have settled to the bottom.

After removal of the acid sludge, the distillate may or may not be treated with a small quantity of strong caustic soda (approximately 30 Baumé) in order to collect any fine particles of sludge which remain suspended in the oil. The distillate is again permitted to settle and the soda and sludge are withdrawn.

The distillate is then neutralized with a weak solution of caustic soda of approximately 3½ Baumé and allowed to settle and any precipitated alkali or sediment is drawn off.

The distillate is then washed free of all soluble impurities. The washing may be

with live steam, or first with water and then with steam, or with water and steam concurrently; as in the washing operation following the removal of the fatty acids.

The distillate is then pumped to a still and a small quantity of absorbent mixed with it. Approximately three per cent of fine 100-200 mesh fuller's earth has been used with good results. The distillate is then reduced with steam and fire until the desired tests on the stock have been obtained. The finished stock may be either pumped through a press or the absorbent may be permitted to settle in the still.

If the distillate, after removal of fatty acids and washing with live steam, is first reduced, then to the reduced distillate is applied the same process as that hereinbefore described up to and including the last steam wash. That is, the reduced distillate is first treated with a mineral acid, then (preferably) with a strong alkali, then with a weak alkali, and then washed with water and steam, or steam alone. The reduced distillate is then allowed to settle and, after the free water has been drawn off, is heated to about 110 to 120° F. and blown free from moisture with air.

While, in claiming the invention, I enumerate lastly the reduction of the distillate to stock, I do not mean to be understood as implying that this step of the process necessarily follows the steps preceding it in the claim, as it is also within my invention to reduce the stock at the stage of the process preceding the acid treatment, as explained in the preceding paragraph.

Having now fully described my invention, what I claim and desire to protect by Letters Patent is:

The process of manufacturing a non-emulsifiable mineral oil of high dielectric strength, which comprises distilling crude petroleum, then treating the distillate with an alkali to saponify the contained fatty acids, then separating out the fatty acid soaps, and then washing suspended matter out of the distillate with live steam; treating the distillate with sulfuric acid and separating out the resultant acid sludge, then again treating the distillate with an alkali and separating out the resultant precipitate, and then again washing the distillate with live steam; and mixing with the distillate fuller's earth after one of the specified treatments with an alkali and reducing the distillate.

In testimony of which invention, I have hereunto set my hand at Phila., Pa., on this eighth day of March, 1923.

HAROLD T. MAITLAND.