Feb. 18, 1930. L. H. CLARK 1,747,161

METHOD OF PURIFYING OIL

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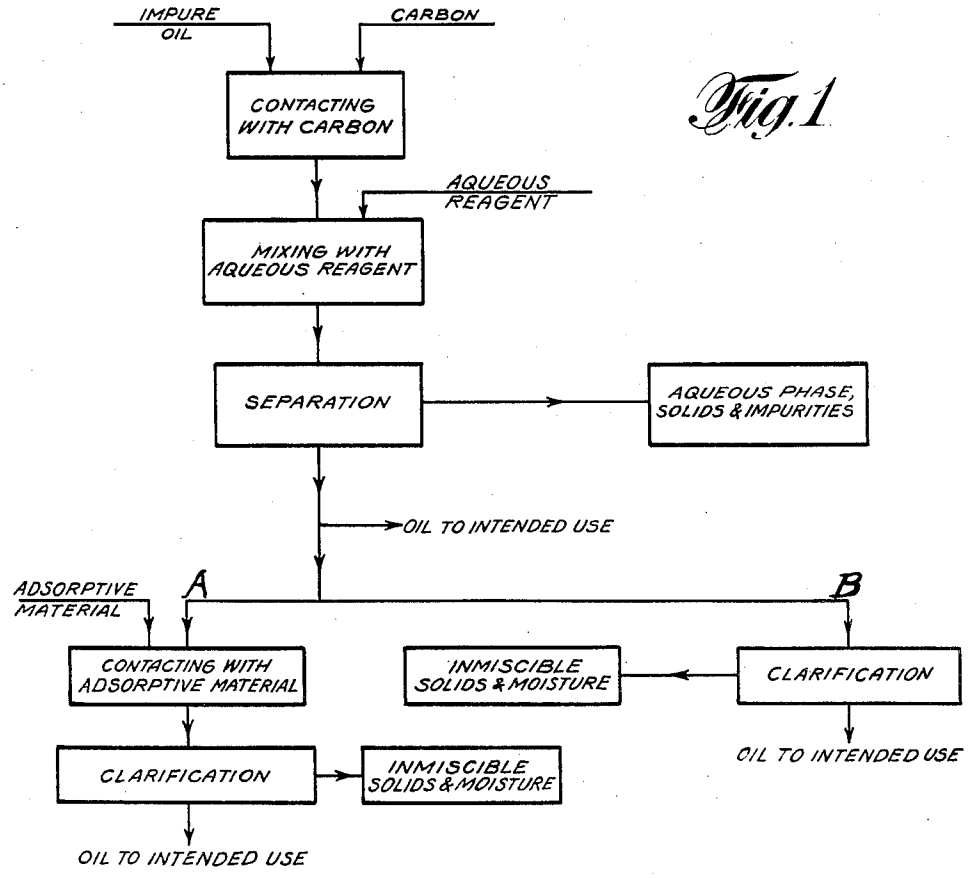
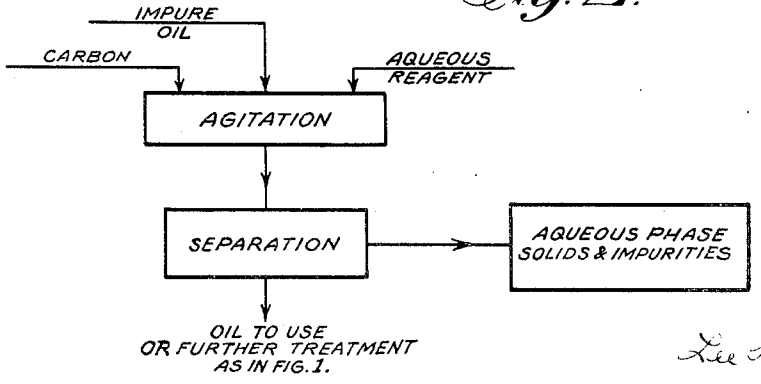


Fig. 1.

Fig. 2.



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UNITED STATES PATENT OFFICE

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METHOD OF PURIFYING OIL

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My invention relates to the purification of oil and particularly to a method for the purification or restoration or renovation of oil that contains such impurities as products of decomposition or chemical change in the oil or some of its constituents, free fatty acids, water, soaps, sludges usually having an acid reaction, some of which are insoluble and others of which are soluble in the oil at temperatures above normal atmospheric temperatures, or sludge-forming substances and other impurities and that may or may not contain finely divided carbon which may be wholly or in part colloidal, such impurities having been introduced into the oil during or by reason of the use made of the oil or otherwise.

An object of my invention is to provide a method whereby oil containing some or all of the above mentioned undesirable substances may be brought to a condition wherein it is well adapted to a particular use.

An important example of oil that contains impurities that are removed in the practice of my invention is oil that has been used to immerse transformer coils. Other examples of oils that may be purified in the practice of my invention are oils that have been used to immerse electric switches and circuit breakers and oil that has been used for the lubrication of internal combustion engines.

In the practice of my invention the resistance of the oil to emulsification may be improved or, if that property of the oil is satisfactory, it may be preserved or remain unimpaired. In order to improve or maintain the resistance of the oil to emulsification it is sometimes desirable in the practice of my invention to take special precautions in carrying out some of the steps of my process; but if it is desirable to merely remove certain impurities from the oil without regard to the ultimate ability of the oil to resist emulsification such precautions may be omitted and in some cases some of the steps of my process as completely set forth herein may be omitted. In any event it is to be understood that with different oils it may be necessary to ascertain by test in accordance with the principles herein set forth what special precautions and what particular steps of my complete invention as herein set forth are to be employed to attain the best results.

Other oils than those specifically mentioned and which contain some or all of the impurities above mentioned, naturally or by reason of the use to which they have been put, may be purified or restored in accordance with my invention; and although purification of oil in the practice of my invention may neutralize or reduce or otherwise remove the acidity of the oil that effect occurs in the purification thereof and my invention is not to be confused with mere neutralization of sour oils that are produced in the course of acid purification in petroleum refining.

Other and further objects of my invention will appear from the following description.

In purifying, in the practice of my invention, oil that for any reason contains one or more of the above-mentioned impurities, the oil is first treated with finely divided activated carbon and then treated with an aqueous reagent having the properties herein set forth and that is preferably alkaline and may in some instances contain a colloidal substance, after which impurities of the oil and the aqueous phase of the resulting mixture are separated from the oil preferably by centrifugal force. If the oil so treated possesses the desired properties no further treatment is necessary but in some cases further purification such as the removal of traces of moisture from the oil may be effected by passing the oil through a centrifugal clarifier; and if it be desired to remove such moisture and additional impurities that impair the resistance to emulsification of the oil that is separated from the aqueous reagent, such results may be effected by treating the oil with an adsorptive medium that is subsequently removed from the oil. As a variation of the foregoing procedure the activated carbon may be brought into proper relation with the oil by being mixed with the aqueous reagent that is subsequently dispersed throughout the oil. Furthermore, dispersion throughout the oil of any of the substances brought into contact with the oil

may be facilitated by reducing the viscosity of the oil as by raising the temperature thereof.

If it be desired to treat the oil with activated carbon before treating the oil with an aqueous reagent, such activated carbon is directly added to and agitated with the oil, the carbon preferably being in a finely divided state and preferably not exceeding 1% by weight of the oil to which it is added. As above stated the viscosity of the oil may be reduced during this operation.

The aqueous reagent, in accordance with my invention, must have the property of being more readily dispersed throughout the oil than substantially pure water and it must have the property of facilitating or causing impurities of the oil to pass from the oil to the interface between the aqueous reagent and the oil or through that interface into the aqueous reagent; and the aqueous reagent preferably has such consistency or composition or such properties that water soluble products of any reaction between the reagent and the oil or its impurities will dissolve in it. The aqueous reagent may consist of an aqueous solution of caustic soda, soda ash, tri-sodium phosphate, or other alkaline substances that produce the desired effect while in solution. In the practice of my invention advantage may be gained by the use of an aqueous reagent of which at least some part of the substance present is in colloidal suspension or solution. An effective solution of such character may be produced by the use of sodium silicate, particularly sodium silicate in which silica predominates by weight, for example in which there are 3.25 parts by weight of SiO_2 to each part of Na_2O, or sodium aluminate or sodium tungstate or any reagent that gives sufficient alkaline concentration and in addition gives naturally, or can be made to give by combination with other agents, the required colloidal condition. This condition is attainable by introducing substances varying from finely divided silica to aluminum hydroxide.

In the practice of my invention good results are attainable by the use of an aqueous reagent having an alkalinity, available under the existing conditions though not in every case initially free, equivalent to that of a solution containing ¼% to 1% by weight of caustic soda, a solution having an alkalinity equivalent to such a ½% solution of caustic soda having been found generally applicable and convenient for commercial work. In any event the degree of alkalinity of the reagent must be such as to render the reagent capable of being easily dispersed in oil and capable of causing removal of impurities therefrom, excessive alkalinity being avoided in order that soaps present in the oil or formed with the fatty acid or acid reacting substances thereof may not be prevented from dissolving in the aqueous phase of the resulting mixture. If, in the practice of my invention, the activated carbon is not added directly to the oil it will be mixed with the aqueous reagent and so introduced to the oil.

In the practice of my invention the aqueous reagent is mixed with the oil preferably by agitation sufficiently violent and prolonged to carry small particles or globules of the reagent to close proximity but not necessarily into contact with every part of the oil that is to be purified. The mixture is advantageously maintained in a heated state, for example 150° F. to 200° F. during the agitation thereof, the temperature being dependent upon the viscosity of the oil.

The aqueous phase and impurities including the activated carbon are then separated from the oil preferably by passing the oil through a centrifugal separator; and, I have found that no process has been suggested by others for purifying oil containing the above-mentioned impurities whereby centrifugal machines may be employed and that no purifying agents heretofore suggested by others make it possible to effect the separation in a centrifugal machine.

The aqueous reagent should be sufficient in quantity and strength to satisfy the affinities of the impurities in the oil and still provide a sufficient excess of active reagent to insure that the reagent will be dispersed throughout the oil and cause the impurities to be removed therefrom as by passing into or dissolving in the aqueous phase of the resulting mixture. The relative quantity of aqueous reagent added to the oil may vary in the treatment of different oils and oils containing different impurities. If the oil that is discharged from the centrifugal separator is found by examination or test to have been sufficiently purified so that either with or without further treatment in accordance with my invention as herein described, it is suitable or may be made suitable for reuse, the alkaline concentration of the reagent and the quantity mixed with the oil is correct. But, if the oil does not appear to be sufficiently purified that is an indication that the conditions of the aqueous phase were incorrect for the particular oil treated. Thus, if the oil discharged from the centrifugal separator appears to be insufficiently purified and the aqueous discharge from the separator is turbid but light in shade, the degree of alkalinity of the reagent was insufficient to effect a substantial removal of the impurities. On the other hand, if the aqueous discharge contains a high concentration of removed impurities and the oil appears to have been insufficiently purified, the mixture produced by the agitation contained an insufficient proportion of the aqueous phase. In the practice of my invention I have found that satisfactory results are at-

tainable if an alkaline reagent of the alkalinity above set forth is mixed with the oil in the proportion of eight to ten and preferably nine parts of contaminated oil to one part of the aqueous alkaline reagent. If the degree of concentration of the aqueous reagent is too high, water soluble products of the reaction between the reagent and the oil or impurities thereof will form a sludge that prevents impurities from passing from oil to the aqueous phase, that cannot be completely separated from the oil either by gravity or centrifugal force, and that carries into the oil impurities that should be removed therefrom. By the use of a reagent having the properties above set forth no sludge layer is formed between the oil and the aqueous phase. It is because all impurities of products thereof that are water soluble are in solution that it is possible to separate the aqueous phase and impurities and sediment from the oil by centrifugal force. Thus, the purification of oil in accordance with my invention makes it possible for the first time to remove impurities of the character above-mentioned by centrifugal force but removal in that manner is not essential to my invention.

Impure oil that has been treated with activated carbon and also treated with an aqueous reagent as above set forth and then passed through a centrifugal separator may be sufficiently purified to meet the requirements of the purpose for which the oil is to be used, but oil that has previously been so treated may be further purified by being passed through a centrifugal clarifier, such clarification removing residual water and other impurities. If the oil discharged from the centrifugal separator in the practice of my invention as above set forth requires further treatment, particularly if the resistance to emulsification of such oil is to be improved, adsorptive material is now added to the oil and agitated therewith and the resulting mixture is then advantageously passed through a centrifugal clarifier. I have found that the addition to oil discharged from the centrifugal separator of only small amounts, of the order ¼% to 1%, of adsorptive material will effect purification and improvement of the resistance of the oil to emulsification that cannot be obtained by the use of even very large amounts of adsorptive material if the oil has not previously been treated in accordance with my invention as above set forth. Treatment of the oil in accordance with my invention as above set forth puts the oil in such condition that such small amounts of adsorptive material will further purify it and effect that purification or alteration of the oil whereby its resistance to emulsification is made satisfactory. If the resistance of the impure oil to emulsification is not particularly poor, treatment of the oil as above described with a reagent containing no colloidal substance will not improve that resistance but after such treatment such small amounts of adsorptive material will make that resistance, even though originally poor, satisfactory. If the resistance of the impure oil to emulsification is originally very poor that resistance is improved when a reagent containing a colloidal component is used and is made entirely satisfactory by further treatment with such small percentage of adsorptive material.

My process above described is particularly effective in the purification of transformer oil, and oils containing sludge, especially sludge that is soluble at increased temperature.

In the treatment of certain oils the results may be improved by delaying the introduction into the clarifier of oil discharged from the centrifugal separator. While the centrifugal separator may be so handled in the operation of separating the aqueous phase impurities and activated carbon from the oil, that the oil discharged from the separator is sufficiently free of moisture, operation of the separator to secure such a result may be less efficient than a less careful handling of the separator followed by centrifugal clarification.

While I have referred only to activated carbon I do not intend to be limited to any particular form of carbon but intend that the use of any carbon that is adsorptive and falls within the class of decolorizing or deodorizing carbons is within the spirit of my invention. While I have found that the use of such carbon in the manner and for the purpose herein set forth produces highly satisfactory results I do not venture any theory in explanation of the manner in which those results are produced.

Purification of oils contaminated as above described cannot be effected by methods heretofore known such as previously known centrifugal processes or filtering devices, except that purification of such oils may be effected as set forth in my co-pending Patent No. 1,553,141 issued September 8th, 1925. And, it will be apparent from the foregoing disclosure that in accordance with my invention oils contaminated as above described may be put into condition for the particular use for which they are desired and the resistance of the oil to emulsification may be improved by the selection of the aqueous reagent used or by treatment with an adsorptive medium after treatment with a suitable aqueous reagent or by both of these treatments, it being possible that the use of activated carbon also improves the resistance of the oil to emulsification. Unless sludge soluble above 100° F. and substances forming soluble sludge are removed, the oil will become cloudy after treatment, and substances ordinarily used for

the purification of oil form with such sludge, or sludge-forming substances, compounds which are highly soluble in the oil and which leave the oil contaminated. However, purifying treatment in accordance with my invention so completely purifies oil containing such substances that a high degree of purity is indicated by even such tests as the tests for resistance to emulsification.

In the practice of my invention the amount of activated carbon added to the oil will ordinarily be less than 1% of the oil by weight but it may be found in practice that additional activated carbon should be used. In the practice of my invention I have found that a suitable aqueous reagent may consist of a solution containing 2% by weight of sodium silicate of the composition above set forth; and that satisfactory results are obtained by mixing such aqueous reagent with the oil in the proportions above set forth. I have also found that caustic soda solution containing ½% of caustic soda will give satisfactory results. With such an aqueous reagent water soluble products of any reaction between the reagent and the oil or any of its impurities, including products of saponification, will be soluble in the aqueous phase and if a mixture of impure oil and such reagent is settled by gravity no layer of sludge will be formed between the oil and the aqueous phase and the entry of finely divided carbon into the aqueous phase is not interfered with. It is because these conditions exist in my process that the aqueous phase and sediment may be separated by centrifugal force. If the oil to be purified contains relatively high percentages of acid impurities it is better to increase the proportion of reagent to the oil than to increase the alkaline concentration of the aqueous reagent.

For the purpose of rendering more quickly apparent the general features of my invention and one mode of operation thereof, there is shown in the annexed drawings one application of my invention to the purification of oil and some of the many possible modifications of that application of my invention. Thus, in the drawings,

Fig. 1 shows schematically one group of steps that may be followed in the practice of my invention; and

Fig. 2 shows schematically a modification of some of the first steps indicated in Fig. 1.

Figs. 1 and 2 may be considered as flow sheets in certain applications of my invention, the branches A and B in Fig. 1 indicating alternative treatments of oil recovered in the separating step, either of which may be applied to such oil after the steps shown in Fig. 2 are carried out.

It is to be understood, however, that my invention is not limited to the general operation or to the modifications thereof shown in the drawings inasmuch as the invention may be embodied in other general operations and may include modifications that are not shown in the drawings. It is also to be understood that my invention is not limited to details thereof or specific applications thereof that are herein described but it is intended that my invention shall include such variations and modifications as fall within the hereunto appended claims.

What I claim as new and desire to secure by Letters Patent of the United States is:

1. The method of purifying mineral oil that contains decomposition products, free fatty acid and sludge or sludge forming substances which comprises contacting such oil with activated carbon and dispersing in the oil an aqueous alkaline reagent containing a uni-valent alkali, and separating from the oil together the activated carbon, impurities and the aqueous phase of the resulting mixture.

2. The method of purifying mineral oil that contains decomposition products, free fatty acid and sludge or sludge forming substances which comprises mixing finely divided activated carbon with such oil and dispersing an aqueous alkaline reagent in said oil containing said carbon and thereby producing an emulsion, and separating from the oil the activated carbon, impurities and the aqueous phase of the resulting mixture.

3. The method of purifying mineral oil which comprises contacting the oil with activated carbon and an aqueous alkaline reagent, separating from the oil the activated carbon, impurities and the aqueous phase of the resulting mixture, and then treating the oil with adsorptive material and thereby improving its resistance to emulsification.

4. The method of purifying mineral oil which comprises contacting the oil with not more than 1% by weight of finely divided activated carbon, dispersing in the oil containing such carbon an aqueous alkaline reagent, and centrifugally separating from the oil together the activated carbon, impurities and the aqueous phase of the resulting mixture.

5. The method of purifying mineral oil which comprises contacting the oil with not more than 1% by weight of finely divided activated carbon, mixing the oil with an aqueous alkaline reagent, centrifugally separating from the oil the activated carbon, impurities and the aqueous phase of the resulting mixture, and then treating the recovered oil with less than 1% by weight of adsorptive material and thereby improving its resistance to emulsification.

6. The method of purifying mineral oil containing decomposition products, free fatty acids and sludge or sludg-forming substances which comprises contacting such oil simultaneously with activated carbon and an aqueous alkaline reagent containing a uni-

valent alkali and separating from the oil together the activated carbon, impurities and the aqueous phase of the resulting mixture.

7. The method of purifying impure mineral oil which comprises contacting the oil with activated carbon and dispersing throughout the oil an aqueous alkaline reagent sufficiently dilute that water soluble impurities and water soluble products of any reaction produced by the reagent will be in solution in the aqueous phase of the resulting mixture, and separating from the oil the activated carbon, impurities and the aqueous phase of the resulting mixture.

8. The method of purifying impure mineral oil which comprises contacting the oil with finely divided activated carbon, dispersing throughout the oil an aqueous alkaline reagent sufficiently dilute that water soluble impurities and products of saponification will be in solution in the aqueous phase of the resulting mixture, and separating from the oil the activated carbon, impurities and the aqueous phase.

9. The method of purifying impure mineral oil which comprises contacting the oil with finely divided activated carbon, dispersing throughout the oil an aqueous alkaline reagent sufficiently dilute that water soluble impurities and water soluble products of any reaction produced by the reagent will be in solution in the aqueous phase of the resulting mixture, separating from the oil activated carbon, impurities and the aqueous phase, and treating the recovered oil with adsorptive material and thereby increasing the resistance of the oil to emulsification with water.

10. The method of purifying impure mineral oil which comprises contacting the oil with finely divided activated carbon, dispersing throughout the oil aqueous alkaline reagent capable of being easily dispersed within the oil and having the property of facilitating the passage of carbonaceous impurities from the oil into the aqueous phase of the resulting mixture and containing in suspension colloidal substance capable of causing carbon particles to lie within the aqueous phase, and separating from the oil the aqueous phase and impurities.

11. The method of purifying mineral oil which comprises contacting the oil with finely divided activated carbon, dispersing throughout the oil an aqueous alkaline reagent containing a substance in colloidal form, and separating from the oil the activated carbon, impurities and the aqueous phase of the resulting mixture.

12. The method of purifying impure mineral oil which comprises contacting the oil with finely divided activated carbon, dispersing throughout the oil an aqueous alkaline reagent containing a colloidal substance in suspension and sufficiently dilute that water soluble impurities and water soluble products of any reaction produced by the reagent will be in solution in the aqueous phase of the resulting mixture, and separating from the oil the activated carbon, impurities and the aqueous phase.

13. The method of purifying mineral oil that contains decomposition products, free fatty acid and sludge or sludge forming substances which comprises contacting the oil with activated carbon and mixing the oil with an aqueous alkaline reagent, separating from the oil together the activated carbon, impurities and the aqueous phase of the resulting mixture, and then treating the oil with adsorptive material.

14. The method of purifying mineral oil that contains decomposition products, free fatty acid and sludge or sludge forming substances which comprises contacting the oil with activated carbon and mixing the oil with an aqueous alkaline reagent, separating from the oil the activated carbon, impurities and the aqueous phase of the resulting mixture, and then treating the oil with not more than one percent by weight of adsorptive material.

15. The method of purifying mineral oil containing decomposition products, free fatty acid and sludge or sludge-forming substances, which comprises simultaneously contacting the oil with activated carbon and dispersing in the oil an aqueous solution of sodium silicate and thereby producing an emulsion containing an aqueous phase, and separating from the oil together the activated carbon, impurities and the aqueous phase of the resulting mixture.

16. The method of purifying mineral oil which comprises simultaneously contacting the oil with not substantially more than one percent by weight of activated carbon and with aqueous alkaline reagent in the proportion of eight to ten parts of oil to one part of aqueous alkaline reagent, and separating from the oil together the activated carbon, impurities and the aqueous phase of the resulting mixture.

17. The method of purifying mineral oil which comprises contacting the oil with activated carbon and dispersing therethrough a substantially two percent aqueous solution of sodium silicate, and separating from the oil together the activated carbon, impurities and the aqueous phase of the resulting mixture.

18. In the purification of mineral oil, the process comprising introducing not substantially more than one percent by weight of activated carbon into oil contaminated by use for submersion of electrical apparatus and dispersing in the oil an aqueous alkaline solution and thereby producing a mixture having an aqueous phase, and separating from the oil together the activated carbon, impurities

and the aqueous phase of the resulting mixture.

19. The method of purifying mineral oil that contains decomposition products including soluble sludge, which comprises contacting such oil with finely divided activated carbon, dispersing in the oil containing such carbon an aqueous alkaline reagent containing a uni-valent alkali, and centrifugally separating from the oil immiscible solids and the aqueous phase of the resulting mixture.

In testimony whereof, I have signed my name to this specification.

LEE H. CLARK.