

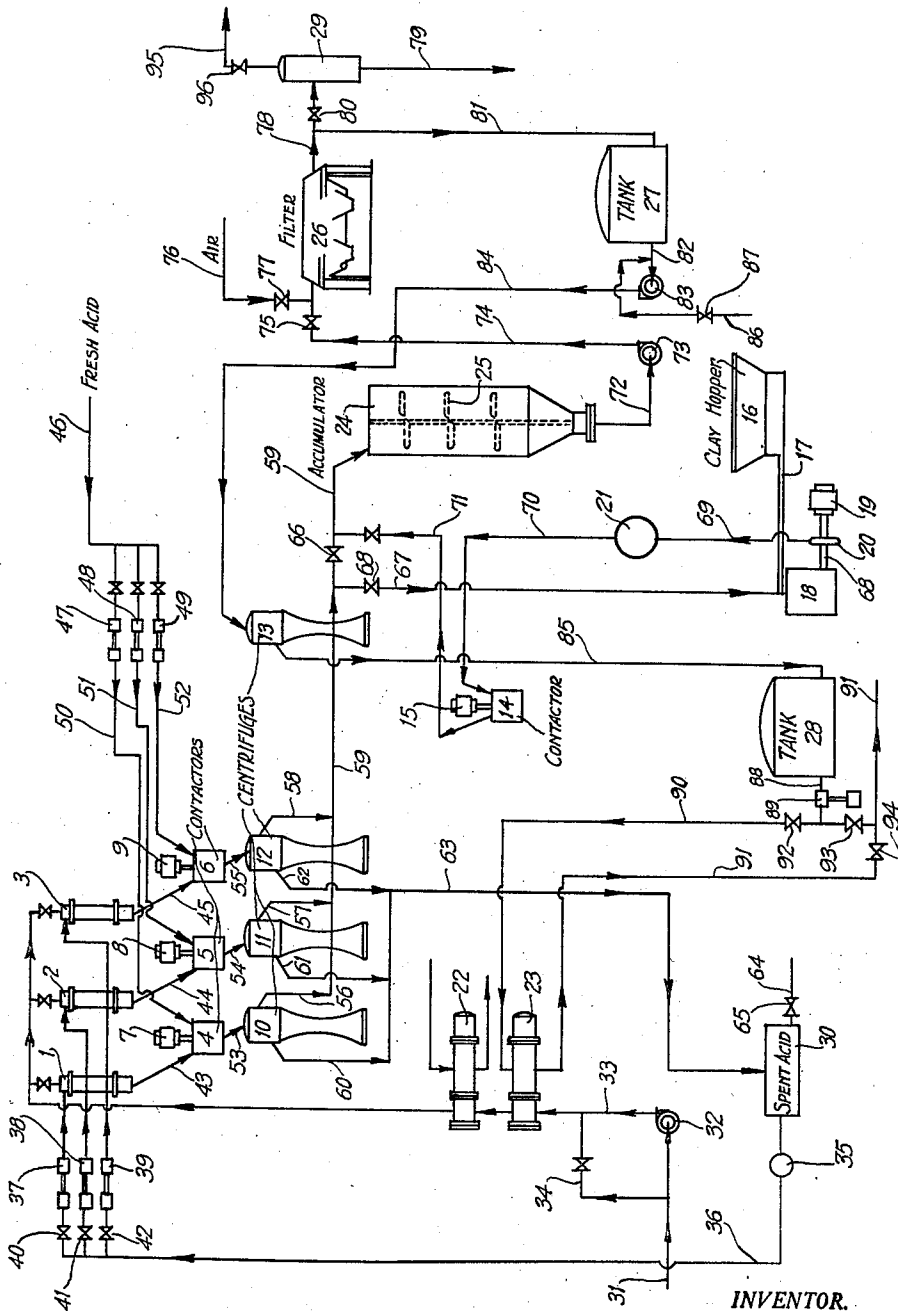
May 9, 1933.

E. PETTY

1,908,616

PROCESS FOR REMOVING IMPURITIES FROM OIL

Filed Jan. 13, 1930



INVENTOR.
Earl Petty.
BY *Thos. E. Seefeld*
ATTORNEY

UNITED STATES PATENT OFFICE

EARL PETTY, OF KANSAS CITY, MISSOURI, ASSIGNOR, BY MESNE ASSIGNMENTS, TO
CROSS DEVELOPMENT CORPORATION, OF WILMINGTON, DELAWARE, A CORPORATION
OF DELAWARE

PROCESS FOR REMOVING IMPURITIES FROM OIL

Application filed January 13, 1930. Serial No. 420,449.

This invention relates to improvements in method and apparatus for treating petroleum hydrocarbons, and refers more particularly to a process for treating either lubricating oils or lighter oils such as gasoline, naphtha, or the lighter distillates of petroleum.

Among the salient objects of the invention are:

10 To provide a process by means of which the asphaltic materials and objectionable sulphur compounds are selectively eliminated from the oil by rapid contact with an acid, with a subsequent immediate separation of the acid and the oil;

15 To provide a process in which the acid treated oil is thereafter intimately mixed with an adsorptive clay which not only has a neutralizing effect to the oil but also selectively removes the reaction products formed and eliminates the loss due to the formation of emulsions and nonsoluble soaps which accompany the neutralizing action where liquid neutralizing agents are employed;

20 To provide a process in which the spent acid may be utilized as a premixing medium to assist and supplement the effect of the fresh acid treatment; and in general, to provide a process of the character hereinafter described in more detail, together with the apparatus used therefor.

25 The single figure is a diagrammatic view of an apparatus which is adaptable to carry out the process explained. Referring to the drawing; the numerals 1, 2, and 3 designate premixers of any suitable type either containing mechanical agitators or utilizing nozzles whereby an intimate contact is obtained between the spent acid and oil. 4, 5, and 6 denote contacting devices by means of which a filming of the acid and oil is produced and a rapid and intimate contact between the oil and acid effected. These contactors are driven by motors shown at 7, 8, and 9. 10, 11, and 12 are centrifugal separators in which the primary separation between the oil and acid is effected. A similar separator is shown at 13, the purpose of which is described during the disclosure of

the operation of the process. Also at 14 is a contactor driven by motor 15, similar to that shown at 7, 8, and 9.

16 is a clay hopper having a mechanical screw feed 17, which discharges the clay into the mixing tank 18. 19 is a motor driving a centrifugal pump or closed impeller 20. 21, 22, and 23 are heat exchangers. 24 is a clay mixer or agitator in which are positioned the mechanical paddles 25. 26 is a filter which may be of any suitable construction. 27 and 28 are receiving tanks and 29 is a gas separator. 30 is a collecting vessel for the sludge.

Describing now the operation of the system; the oil to be treated is introduced from any convenient source through the inlet line 31 and is forced by means of a pump 32 through the line 33 and heat exchangers 23 and 22 into the separate premixers 1, 2, and 3. A by-pass 34 is shown around the pump 32.

Spent acid collected from the first contacting operation accumulated in the sludge tank 30 is recycled therefrom by means of the pump 35 and pipe 36 and distributed by means of the separate pumps 37, 38, and 39 to the separate premixing stages 1, 2, and 3 respectively, through suitable pipe lines. Valves 40, 41 and 42 are interposed in the suction lines of the respective pumps in order that any particular stage may be controlled or cut out of the system as desired. In the premixing stages the oil and spent acid are contacted and the separate mixtures pass through the pipes 43, 44, and 45 to the contactors 4, 5, and 6, where the mixtures are first subjected to fresh acid supplied from the fresh acid source through pipe 46 and separate charging pumps 47, 48, and 49, the discharge lines 50, 51 and 52 connected to the respective pumps discharging into the contactors 4, 5, and 6 respectively. Due to the peculiar construction of the contactors a very rapid interfacial mixing or filming of the acid and oil is effected therein, after which the mixed acid and oil is discharged through the pipes 53, 54, and 55 into the centrifugal separators 10, 11, and 12. In the separators, the oil and acid are separated, the former be-

ing discharged through pipes 56, 57 and 58 into a common line 59, while the acid is drawn off through pipes 60, 61, and 62, thence through a common line 63, to the sludge accumulator 30. A separate drawoff line 64 is connected to the sludge tank and controlled by valve 65, by means of which spent acid which is not recycled may be diverted from the system and in order that the recycled acid concentration is maintained uniform.

Interposed in the pipe 59 is a valve 66 which, when closed, diverts the oil through the line 67 controlled by the valve 68. The line 67 discharges into the clay mixing tank 18 where the oil and clay are mixed. From this tank the mixture of oil and clay is withdrawn through a pipe 68 and is forced by means of the pump 20 through a pipe 69 and heat exchanger or heater, preferably heated by steam or by bringing the mixture in contact with a hotter medium such as the hot oil.

From the heat exchanger the oil is directed through a line 70 to the contactor 14, where an intimate mixture of the clay and oil is produced. Being discharged from the contactor the mixture passes through the pipe 71, thence again into that portion of the pipe 59 beyond the valve 66, the latter discharging into the clay accumulator tower 24. The function of the accumulator 24 is to furnish a place to collect the mixed clay and acid treated oil, to prevent overloading the filter and to permit the filter being cut out from time to time for cleaning. The discharge pipe 72 connected into the bottom of the accumulator is connected to the suction side of the pump 73 by means of which the mixture is directed through a line 74 controlled by a valve 75, to the filter 26.

An air line 76 regulated by valve 77 provides a means for maintaining proper pressure upon the filter. Within the filter the clay is removed from the oil and the latter passes off through the discharge line 78 and may be directed immediately to the gas separator 29, thence to naphtha storage through the pipe 79 or, by closing the valve 80, the oil will pass through the line 81 to the receiving tank 27. A withdrawal line 82 from the receiving tank directs the oil to the pump 83 by means of which it is forced through a pipe 84 and subjected to centrifugal separation in the centrifuge 13, after which the clarified product passes through the line 85 to the receiving tank 28.

When necessary, a final neutralization may be effected by introducing ammonia or any other desired neutralizing agent into the pipe 82 through a line 86 controlled by a valve 87. The distillate collected in the receiving tank 28 may be drawn off through the line 88 and forced by means of the pump 89 through the pipe 90 and utilized as a pre-heating medium in the heat exchanger 23. On discharge from

the heat exchanger, the oil passes through the line 91 to storage. Valves 92 and 93 are interposed in the line 90, and valve 94 in the line 91 for diverting the oil directly from the receiving tank to storage, or controlling the amount of oil utilized as a heating medium in the exchanger 23 as desired.

A gas outlet pipe 95, equipped with a valve 96 is connected into the top of a gas separator 29. As suggested, the treating method may be used either for the acid treatment of lubricating stock or for motor fuel distillate, including the complete range of overhead products of petroleum which contain objectionable impurities and excessive amounts of sulphur compounds. The process permits the use of much smaller quantities of acid than the usual process in which oils and acids are agitated with air and, being an entirely closed system in which oxygen and air are entirely excluded, the production and formation of materials formed by a secondary reaction of the oil and the acid and oxygen are almost entirely eliminated. The quick and complete contacting of the oil effectively removes objectionable compounds which are subsequently separated by centrifuge and the adsorptive effects of the clay treatment.

Recirculation of the spent acid reduces materially the fresh acid requirements and gives a more complete effect to the fresh acid treatment. Furthermore, only sufficient clay is used to neutralize the acid present and there is a considerable economy of clay as compared to normal clay contacting treating methods.

It will be appreciated that, with different oils, different requirements are necessary as to the acid treatment and subsequent clay treatment but, in every case, comparative figures show a marked advantage in the economy of the present method. An improved final product is also obtained due to the fact that considerably lower temperatures are present particularly in the contacting stage as compared to normal air agitation methods.

In a typical example, a lube oil stock is contacted with 66° Beaumé sulphuric acid for a period of from thirty seconds to three minutes, during which period secondary reactions or reabsorption of the sludge from the first reaction back into the acid oil was strictly eliminated. The time factor will, of course, vary with the character and concentration of the acid and the extent of the recirculation of the spent acid. Where spent acid recirculation is not used, the time factor is considerably more than when recirculation is employed. The temperature of the oil itself is a factor in the necessary time for contacting. Viscous lubricating oils require considerably greater acid contacting time than the lighter distillates such as naphtha.

To neutralize the acid with an adsorptive material such as clay after the contacting and centrifugal separation, only sufficient clay is

used which is necessary to neutralize the acid present. Normally, six pounds per barrel or less have been sufficient as compared with a minimum of eighteen pounds necessary in the usual air agitation methods. Where a pressure mixer is used with a small amount of clay, it may be desirable to dilute the oil in place of relying upon the steam heating of the mixture to produce the workable viscosity. Such dilution may be resorted to where it is undesirable to carry the temperatures as high as would be necessary to procure workable viscosity. By utilizing clay as a neutralizing agent in place of a liquid neutralizer and water, the loss produced by the formation of emulsions and soluble soaps or sulphates is avoided and all reacting materials absorbed upon the clay so that they can be mechanically removed in place of relying upon the settling action to remove the reaction materials after neutralizing in connection with liquid neutralizers. The final separation or filtration may be made in any suitable type of filter or by means of centrifugal clarifiers.

I claim as my invention :

1. A process for removing impurities from oil comprising the steps of mechanically mixing fresh acid with the oil to effect a rapid and complete contact therewith, immediately separating the oil and acid by centrifugal separation, contacting the acid treated oil with an adsorptive clay, separating the clay from the oil and utilizing the spent acid recovered for pretreating the oil prior to its contact in the fresh acid treating step, said fresh acid treating step being then carried on in the presence of the spent acid.

2. A process for removing impurities from oil comprising the steps of mechanically mixing fresh acid with the oil to effect a rapid and complete contact therewith, immediately separating the oil and acid by centrifugal separation, contacting the acid treated oil with an adsorptive clay, separating the clay from the oil and utilizing the spent acid recovered for pretreating the oil prior to its contact in the fresh acid treating step, said fresh acid treating step being then carried out in the presence of the spent acid, and subjecting the final treated oil to neutralization with ammonia.

3. A process for removing impurities from oil comprising the steps of pretreating the oil and mechanically mixing fresh acid with the oil to effect a rapid and complete contact therewith, immediately separating the oil and acid by centrifugal separation, contacting the acid treated oil with an adsorptive clay, separating the clay from the oil and utilizing the spent acid recovered for pretreating the oil prior to its contact in the fresh acid treating step, said fresh acid treating step being then carried on in the presence of the spent acid.

4. A process for removing impurities from oil comprising the steps of mechanically mixing fresh acid with the oil to effect a rapid and complete contact therewith, immediately separating the oil and acid by centrifugal separation, contacting the acid treated oil with an adsorptive clay, separating the clay from the oil, utilizing the separated oil for preheating the oil to be purified and utilizing the spent acid recovered for pretreating the oil prior to its contact in the fresh acid treating step, said fresh acid treating step being then carried on in the presence of the spent acid.

5. A process for removing impurities from oil comprising the steps of mechanically mixing fresh acid with the oil to effect a rapid and complete contact therewith, immediately separating the oil and acid by centrifugal separation, contacting the acid treated oil with an adsorptive clay, separating the clay from the oil, utilizing the separated oil for preheating the oil to be purified, utilizing the spent acid recovered for pretreating the oil prior to its contact in the fresh acid treating step, said fresh acid treatment being then carried on in the presence of the spent acid, and subjecting the final treated oil to neutralization with ammonia.

In testimony whereof I affix my signature.
EARL PETTY.