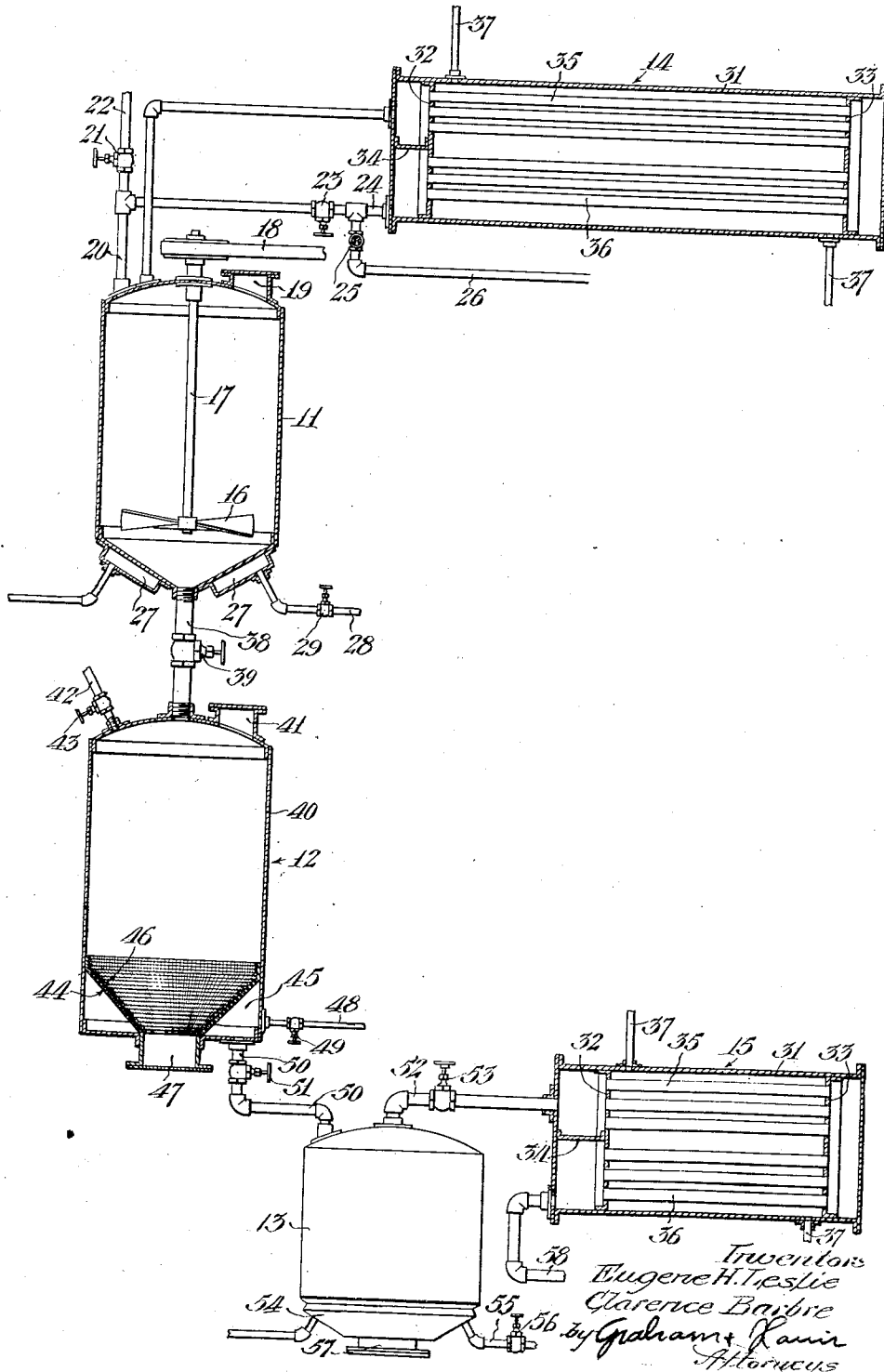


E. H. LESLIE AND C. BARBRE,
 PROCESS OF REFINING CRACKED OILS.
 APPLICATION FILED SEPT. 13, 1917.

1,337,523.

Patented Apr. 20, 1920.



Inventors
 Eugene H. Leslie
 Clarence Barbre
 by Graham & Rair
 Attorneys

UNITED STATES PATENT OFFICE.

EUGENE H. LESLIE AND CLARENCE BARBRE, OF LOS ANGELES, CALIFORNIA,
ASSIGNORS TO GENERAL PETROLEUM CORPORATION, A CORPORATION OF
CALIFORNIA.

PROCESS OF REFINING CRACKED OILS.

1,337,523.

Specification of Letters Patent.

Patented Apr. 20, 1920.

Application filed September 13, 1917. Serial No. 191,131.

To all whom it may concern:

Be it known that we, EUGENE H. LESLIE and CLARENCE BARBRE, both citizens of the United States, residing at Los Angeles, in the county of Los Angeles and State of California, have invented a new and useful Process of Refining Cracked Oils, of which the following is a specification,

Our invention relates to the art of refining petroleum oils, and the principal object of the invention is to provide a process by which a cracked oil may be refined to improve its color and odor and render it more stable. A number of processes have been developed in which petroleum oils are subjected to heat under atmospheric or higher pressures, for the purpose of changing the physical and chemical properties of the oil, the result generally being a treated oil, part of which is more volatile than the raw oil. Such treated oils are ordinarily termed "cracked oils" and they are objectionable for many purposes on account of containing unsaturated and olefinic hydrocarbons as well as unsaturated and bad odored nitrogen and sulfur compounds. In addition such oils often contain organic acids or bases, phenols, and other compounds which tend to give objectionable color and odor. All these materials are hereinafter referred to as "unsaturated compounds" and the term "cracked oils" is herein used as inclusive of all hydrocarbon oils containing such unsaturated compounds. Such cracked oils may be gasolenes, burning, lubricating, or other oils of any specific gravity or viscosity found in the refining art.

By our invention the unsaturated compounds are in part polymerized and condensed and a water white distillate of good odor is produced, from five to fifteen per cent. of the oil treated appearing, however, as a resinous still residue. This resinous still residue is a valuable commercial product and its production is made the subject of a companion application Serial No. 191,198, filed September 25, 1917, entitled "Process of producing synthetic resins."

Further objects and advantages will be made evident hereinafter.

In the drawing, which is very diagram-

matic, an apparatus adapted to carry on our process is shown.

This apparatus consists of a digester 11, a filter 12, a still 13, a primary condenser 14, 55 and a final condenser 15. The digester 11 is provided with an agitator 16, operated through a shaft 17 by means of a belt 18 from a prime mover, not shown. A pipe 20 is secured in the top of the digester 11, being connected through a valve 21 with a cracked oil supply pipe 22, and through a valve 23 with a condensate discharge pipe 24 of the primary condenser 14. The pipe 24 is also connected through a valve 25 with a primary product delivery pipe 26. A charging opening 19, closed by a suitable cover, is provided in the top of the digester. A steam jacket 27, supplied with steam through a pipe 28, controlled by a valve 29, 70 is provided for the purpose of heating the bottom of the digester 11. A vapor pipe 30 carries the vapor from the top of the digester 11 to the primary condenser 14.

The condensers 14 and 15 may be of any 75 suitable design, those shown being identical as to construction and consisting of a closed shell 31, having intermediate heads 32 and 33. A baffle 34 divides the space at the entering end of the condenser into two 80 chambers, the vapor being delivered into the top chamber and passing through tubes 35 into the space between the head 32 and the end of the shell, and through tubes 36 into the lower chamber, from which the condensed products may be removed through a delivery pipe. Water is supplied and removed from the condensers through pipes 37, this water surrounding the tubes 35 and 36 and providing the cooling medium. 90

A pipe 38, controlled by a valve 39, connects the lower portion of the digester 11 with the top of the filter 12. This filter consists of a shell 40, having an opening 41, closed by a suitable cover in the top 95 thereof and having a vapor pipe 42, leading to a condenser, not shown, controlled by a valve 43, also secured in the top thereof. Secured in the bottom is a perforated diaphragm 44 forming with the bottom of the 100 shell 40 a filtered oil space 45. When in operation a canvas or other suitable filter

cloth 46 is placed over the diaphragm 44. A catalyzer discharge opening 47, closed by a suitable cover, is formed in the extreme bottom of the filter 12. A steam blow-out pipe 48, controlled by a valve 49, communicates with the space 45 and an oil discharge pipe 50, controlled by a valve 51, also communicates with the space. The pipe 50 discharges into the top of the still 13, which is provided with a vapor outlet 52 connecting through a valve 53 with the condenser 15. The still 13 is further provided with a steam jacket 54, supplied with steam through a pipe 55, controlled by a valve 56. A residuum discharge opening 57, closed by a suitable cover, is provided in the bottom of the still 13. The final condenser 15 is of the same general construction as the primary condenser 14 and is provided with a final product delivery pipe 58.

The method of operation is as follows:

The cracked oil to be treated is supplied to the digester 11 through the pipe 22, the valve 21 and the pipe 20, the valve 21 being open and the valve 23 being closed. The digester is also charged with a porous catalyzer through the opening 19. The term "porous catalyzer" as used throughout this specification refers to any suitable comminuted material such as fullers' earth, kieselguhr, clay, charcoal, powdered coke, or the like.

The digester may also be charged with a neutralizer. The term "neutralizer" as used in this specification refers to calcium carbonate, other alkaline earth carbonates, weak acid salts of any of the alkali or alkaline earth metals, or to any other suitable metallic base weaker than alkaline earths. The cracked oil, porous catalyzer and the neutralizer are stirred by the agitator 16, heat being applied by the steam jacket 27 on the bottom thereof. We have found that the preliminary boiling may advantageously continue from two to five hours. During this time the valves 21 and 25 are closed and the valve 23 is open so that the vapor passing through the pipe 30 is condensed in the primary condenser 14, the condensate being returned to the digester 11 through pipes 24 and 20. At the end of this time the valve 23 is closed and the valve 25 is opened and from five to twenty per cent. of the oil may be distilled off, passing through the pipe 30 into the condenser 14, where it is condensed, and delivered as a primary product through the primary product delivery pipe 26. After the five to twenty per cent. of oil, above referred to, has been distilled off, it is found that the boiling point will have risen and that it is possible to raise the temperature of the refluxing mass in the digester. This increases the speed of the polymerizing reactions. The

refluxing is now continued for from two to five hours longer with a further production of vapors which are condensed in the condenser 14 and returned through the pipes 24 and 20 to the digester 11, the valve 23 being open and the valve 25 being closed. At the end of this time the valve 23 is closed and the valve 25 opened, and from five to twenty per cent. of the contents are distilled off, being delivered as primary product through the pipe 26. As a result the boiling point of the oil in the refluxer is again higher, and the refluxing is again continued at a higher temperature for several hours more.

The residue is then discharged through the pipe 38 into the filter 12. The liquid components of this residue then pass through the diaphragm 44 and the filter cloth 46 into the space 45. The solid constituents of the residue are removed through the discharge opening 47 after the adhering oil has been distilled off by introduction of steam at 48 and removal of the vapor at 42. The liquid components are delivered through the pipe 50 into the still 13 where they are evaporated, the vapors passing through the pipe 52 into the condenser 15, the condensate from which is a water white distillate which is free from objectionable compounds and without objectionable odor, and which is delivered as a final product through the pipe 58. The residue left in the still 13 is a resinous material, having a considerable commercial value, which may be removed from the discharge opening 57.

The function of the porous catalyzer is to produce a rapid polymerization and resinification of the unsaturated and olefinic hydrocarbon portions as well as the unsaturated and bad odored nitrogen and sulfur compounds. The function of the neutralizer is to remove organic acids, phenols and other compounds which tend to render the oil acid or give it a bad odor.

We claim as our invention:—

1. Any process of refining cracked oils and producing a synthetic resin which includes heating the oil in the presence of a porous catalyzer; filtering out the catalyzer; and distilling the residue to separate the oil from the resins formed by the process.
2. Any process of refining cracked oils and producing a synthetic resin which includes heating and agitating the oil in the presence of a porous catalyzer; filtering out the catalyzer; and distilling the residue to separate the oil from the resins formed by the process.
3. Any process of refining cracked oils and producing a synthetic resin which includes heating the oil in the presence of a porous catalyzer and a suitable neutralizing agent; filtering out the catalyzer; and distil-

ling the residue to separate the oil from the resins formed by the process.

4. Any process of refining cracked oils and producing a synthetic resin which includes heating and agitating the oil in the presence of a porous catalyzer and a suitable neutralizing agent; filtering out the cata-

lyzer; and distilling the residue to separate the oil from the resins formed by the process.

In testimony whereof we have hereunto set our hands at Los Angeles, California, this 8th day of September 1917.

EUGENE H. LESLIE.
CLARENCE BARBRE.