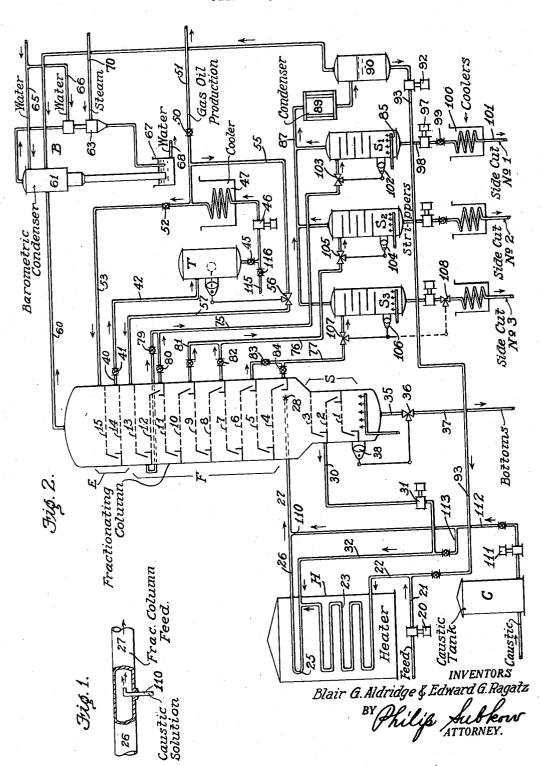
PROCESS FOR PREVENTING CORROSION IN THE DISTILLATION OF HYDROCARBON OIL Filed July 10, 1934



UNITED STATES PATENT OFFICE

2,105,874

PROCESS FOR PREVENTING CORROSION IN THE DISTILLATION OF HYDROCARBON OIL

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Application July 10, 1934, Serial No. 734,529

3 Claims. (Cl. 196-35)

This invention relates to fractional distillation of oil, and particularly to vacuum distillation of hydrocarbon oils to produce lubricating oil distillates.

It is an object of this invention to provide a method of control which will automatically take care of variations in heat and feed input to a fractional distillation system.

It is another object of this invention to 10 eliminate the troublesome tubular oil condenser ordinarily employed for condensation of the overhead fractionated vapors and to substitute therefor a means of condensing the overhead vapors by directly contacting them with cooled recycled oil in a heat exchange tray section in communication with the fractionating system.

It is another object of this invention to provide a method for initially establishing operating conditions within a system which utilizes a heat exchange section associated with the fractionating column in which condensation of the vapors is accomplished by direct contact with cooled oil.

It is another object of this invention to increase the distillation capacity of the distillation system and to avoid undesirable decomposition of the heated feed oil by recirculation therewith through the heater of a quantity of unvaporized oil from the flash section of the fractionating column.

or substantially eliminate the corrosion in the heating system normally attendant upon the caustic solution treatment of the heated feed materials.

This invention resides in a process and apparatus for the fractional distillation of hydrocarbon oils to produce lubricating oil distillates wherein the fractionated vapors are totally condensed by directly contacting them with a cooled 40 circulating oil, and wherein control of the fractionating system is maintained to allow for variations in quantity of heat and feed input by automatically returning to the fractionating column a quantity of reflux sufficient to com- $45\,$ pensate for this variation, and wherein the distillation capacity of the distillation system is substantially increased by recirculating through the heater with the feed a quantity of unvaporized oil from the flash section of the fractionating 50 system, and wherein corrosion of the distillation system, and particularly the heater, is reduced or substantially eliminated by the introduction of caustic solution into the heated feed at a point between the heater and the flash section of the ; fractionating system, whereby the caustic solution is permitted to flash out of contact with the heater surfaces and whereby the thus flashed caustic is returned through the medium of the recirculating oil from the flash section of the fractionating column to the heater.

Figure 1 is an enlarged diagrammatic view of the connections for introducing caustic solution into the system.

Figure 2 comprises a diagrammatic illustration of the preferred apparatus by which the process 10 of this invention is carried on, in which H is a heater for providing a heated oil feed to the fractionating column, S, including plates 1, 2, and 3, is the stripping section, F, including plates 4 to 13 is the fractionating section, E, including 15 plates 14 and 15, is the heat exchange section, and the space between plates 3 and 4 into which the feed is introduced is the separating or flash section. B illustrates a barometric condenser and steam ejector system for maintaining a vacuum 20 in the fractionating and stripping system. T is a surge tank with a float control therein for accumulating and automatically regulating the return of reflux to the fractionating system. SI, S2, and S3 are stripping columns in which inter- 25 mediate distillate cuts received from the fractionating system are stripped of their light components to produce stripped lubricating oil distillates. C is a caustic storage tank from which caustic solution is withdrawn and introduced into 30 the heated hydrocarbon feed to the fractionating column.

The operation is as follows:

Preheated lubricating oil stock feed is introduced by means of pump 20 through lines 21 and 35 22 into the convection section of the heating tubes 23 of the heater H. The partially heated feed is finally heated in the radiant tubular section 25 in the heater H and from there is introduced into the flash section of the frac- 40 tionating column through lines 26 and 27 and inlet 28. The thus heated oil falls upon and flows across plate 3 where it is intimately contacted with rising steam and hydrocarbon vapors from the stripping section S therebelow. Upon 45 thus contacting the said steam and vapors the heated oil feed is partially flashed into vapors which in turn pass upward through the fractionating section F of the fractionating column in countercurrent contact with reflux condensate 50 resulting in the fractionation of the said vapors.

A portion of the unvaporized oil from the flash section of the fractionating column is withdrawn at 30 and reintroduced by means of pump 31 and line 32 into the heating section 25 where it meets

and mixes with the partially heated feed from heating section 23. The mixture of the new heated lubricating oil feed stock and the recirculated oil from the flash section of the column thus constitutes the feed introduced to the said lines 26 and 27 and inlet 28.

The unvaporized oil not withdrawn through 30 from the flash section continues downward over trays 2 and 1 of the stripping section S in 10 counter current to steam to further strip light components therefrom, and the residual oil resulting therefrom is withdrawn through outlet 35, float control valve 36 and bottoms disposal line 37. A float control 38 serves to regulate 15 valve 36 to maintain a constant residuum liquid level in the bottom of the stripping section S.

The fractionated vapors leaving the top trays 13 of the fractionating section F of the fractionating column pass into the heat exchange section 20 E comprising plates 14 and 15 where the said vapors are substantially totally condensed in contact with cooled recycled gas oil. The cooled recycled gas oil containing the commingled condensate formed in the heat exchange section E is 25 withdrawn from plate 14 through outlet line 40, valve 41 and line 42 to the surge tank T from which it is withdrawn through valve 45 and forced by means of pump 46 through the cooler The flow of the thus cooled gas oil is split 30 in three ways, a predetermined quantity controlled by valve 50 being withdrawn to production through line 51, a predetermined constant quantity determined by the setting of valve 52 being returned through line 53 to the heat exchange $35\,$ section E to contact and condense the vapors from the fractionating section of the column as hereinbefore disclosed, and the balance of the gas oil being returned through lines 55 and 57 as regulated by the float control valve 56 for reflux to 40 the top of the fractionating section F.

The air, fixed gases and entrained oil vapors are withdrawn from the top of the fractionating column through line 60 to the condensing system B in which a barometric condenser 61 is provided for condensing the steam and entrained vapors and a steam ejector 63 is provided for exhausting the said barometric condenser of the air and fixed gases. Water, preferably salt water, is supplied to the barometric condenser and the steam ejec-50 tor through lines 65 and 66. The water withdrawn from the barometric condenser leg, and the exhaust of the steam ejector is withdrawn from the tank 67 through outlet 68. Steam is supplied to the steam ejector through steam line

₅₅ 70. Intermediate side cuts of descending reflux condensate are withdrawn from the fractionating section F of the column through lines 75, 76, and 11 to stripping columns SI, S2 and S3 respec-60 tively. The trays from which the said withdrawn side cuts are taken are regulated by the valves 79 and 80 for stripper No. 1, 81 and 82 for stripper No 2, and 83 and 84 for stripper No. 3. Other connections may be provided so that side cuts 65 may be taken from any of the trays in the column desired. The thus withdrawn side cuts are stripped of their light constituents in countercurrent contact with steam which is introduced at the bottoms of the stripping columns as shown 70 at 85 in column SI. The combined stripped vapors and steam pass from the stripping columns through line 87 into the condenser 89 and the resulting condensate collected in receiver 90. The condensate from receiver 90, is returned by 75 means of pump 92 through line 93 and returned

to the feed stream entering the heater through line 22 and is reintroduced therewith into the fractionating system. The fixed uncondensed gases from receiver 90 are exhausted by means of the barometric condenser system B\through 5 line 95 which at the same time maintains the side cut stripping system under vacuum.

The stripped side cuts are withdrawn from the bottoms of the stripping columns to storage and constitute the untreated lube oil distillates. For 10 example, the stripped bottoms from stripping column Si is withdrawn by means of pump 97 through outlet 98 through valve 99, cooler 100, and finally through line ioi to storage. The rates of withdrawal of the bottoms from the 15 stripping columns are regulated by the bottoms valves to a predetermined constant quantity. Float controls 102, 104 and 106 act upon valves 103, 105, and 107 respectively to regulate the quantity of side cut withdrawn from the frac- 20 tionating column to the stripping columns SI, S2, and S3 respectively to maintain constant liquid levels in the bottoms of the said stripping columns.

In some cases where deep cuts of lubricating oil $\,25$ distillate are desired, the control of the withdrawal of condensate from the column to the stripper S3 may be transferred from valve 107 to valve 108. With this arrangement valve 107 is maintained open and all of the descending reflux 30 condensate is withdrawn from the lower end of the fractionating column to the said stripping column S3, and valve 108 is controlled by float control 106 to remove the stripped oil as fast as it is received in the bottoms of the stripper. In 35 this case no reflux condensate is returned to the flash section or stripping section of the column.

It has been found to be advantageous to circulate heavy oil through the heater with the feed material to the extent of one and one-half to 40 three times the feed rate primarily, other conditions being established, to maintain the desired temperature of the flash tray. In recirculating the said unvaporized heavy oil through the heater it is advantageous to withdraw, for that purpose, the unvaporized oil from the flash tray in the bottom of the flash section of the column, which in this case is the oil from the top tray 3 of the stripping section S. This oil from the flash tray is preferred to column bottoms or other $_{50}$ bottoms material for the reason that it is very nearly the equilibrium liquid of the flash section vapors. When this condition obtains, the absorption oil effect of this oil recirculated with the feed stock through the heater is a minimum, per- 55 mitting the distillation of the net overhead vapor cut at a lower temperature than would be possible where column bottoms or other oil less nearly in equilibrium with the vapors were employed.

When hydrocarbon oils are heated to high tem- 60 peratures, especially in the hotter portions of the heater system, naphthenic acids, hydrogen sulphide and other materials are formed which are corrosive to the heater surfaces. In order to eliminate corrosion resulting from the formation 65 of these corrosive substances in the heated oil, it has heretofore been the practice to introduce a quantity of a caustic solution into the feed material entering the heater. It has been discovered, however, that at the point in the heater 70 where the excess water in the said caustic solution flashes, an excessive amount of corrosion takes place. It has been found that this difficulty can be overcome by introducing the caustic solution into the heated feed a short distance 75

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from the entrance to the fractionating column as shown at 110. The feed line to the fractionating column at this point is a large one and it has been found practicable to introduce the small amount of caustic solution necessary by means of a special connection as shown in Figure 2 into the central portion of the feed stream flowing therethrough. Apparently with this arrangement and due to the high velocity of the feed materials at this point and the size of the connection, the thus introduced caustic solution is able to flash entirely within the oil stream without contacting any of the feed line or fractionating equipment surfaces.

15 A large percentage of this flashed caustic solution is retained in the unvaporized oil withdrawn from the flash section of the fractionating column and this material is then introduced into the heating system by way of the hereinbefore described recirculating oil which is withdrawn from the flash section and returned to the heater. Thus only preflashed caustic solution is allowed to enter the heater tubes.

The caustic solution to be introduced into the heated feed is withdrawn from the caustic supply tank C by means of pump !!! and delivered through line !!2 to the point of introduction !!0.

An auxiliary by-pass connection 113 is provided for introducing caustic solution directly into the heater by way of line 32 if desired.

Normally, control of the distillation system is accomplished, as hereinbefore stated, by withdrawing predetermined fixed quantities of gas oil and side cuts to production, allowing the quantity of reflux returned to the column and the quantity of bottoms withdrawn from the fractionating column to vary with variations of input of heat and quantity of feed to the fractionating system. For example, the valve 50 is manually regulated to allow a predetermined quantity of gas oil to be withdrawn to production and the valves corresponding to valve 99 on the strippers adjusted for the withdrawal of predetermined quantities of stripped side cuts, and the quantity of feed introduced into the heater through the feed line 21 and the temperature of the heated feed introduced into the fractionating system through inlet 28 is maintained manually to approximately meet these arbitrarily set conditions. The quantity of recirculated cooled gas oil to the heat exchange trays is also regulated and fixed by means of valve 52. Any irregularity in the quantity and temperature of feed is then automatically compensated for by corresponding vari-55 ations in the quantity of reflux returned to the top of the fractionating section of the fractionating column and the quantity of bottoms withdrawn from the stripping section of the fractionating column. When, due to irregularity of firing of the heater, or for any other reason, the temperature of the feed to the fractionating column is increased, the attendant increase in overhead vapors will result in a greater quantity of fractionated vapors being condensed in the heat exchange section of the fractionating column. This increased quantity of condensate withdrawn from the heat exchange trays along with the recirculating cooling gas oil, will result in an increased accumulation thereof in tank T, since the quantity of gas oil withdrawn to production and the quantity of gas oil recirculated to the heat exchange section of the column is a predetermined fixed quantity. This increase in quantity of condensate accumulating in surge tank T will result in a rise in the liquid level and will act

through the float control therein to regulate valve 56 to allow, in turn, a correspondingly greater quantity of condensate to be returned for refluxing to the top of the fractionating section F of the fractionating column. Thus variations of heat input to the column will automatically be compensated by corresponding variations in quantities of heat absorbing reflux.

The difficulties normally associated with an overhead tubular oil condenser for the condensa- 10 tion of fractionated vapors have been eliminated by the employment of the set of heat interchanger trays as shown at 14 and 15 in the top of the column as hereinbefore described. In this section of the column substantially total conden- 15 sation of the fractionated vapors occurs by contacting them with cool recycled oil. This condensation is accomplished by the exchange of the latent heat of vaporization of the said fractionated vapors for the sensible heat of the said re- 20 cycled oil. The condensate commingles with the oil in the heat exchange section of the column and is withdrawn therewith from the column. No fractionation occurs in this section of the column as evidenced by the fact that the quality 25 of the liquid leaving the lower interchanger tray is substantially the same as that of the vapors entering the tray. In other words, the recycled cooling oil, the condensate resulting from the condensation of the fractionated vapors, the reflux returned to the top of the fractionating column and the gas oil withdrawn to production are all of the same characteristics and quality.

In initiating operations of the fractionating system it is necessary that a cooling medium be 35 supplied to the heat exchange section of the column in order to establish initial condensation of the vaporized feed material, otherwise the vapors may pass through and out of the column without condensation or fractionation. For this 40 purpose a connection 115 with valve 116 is provided for making connection to an outside supply of gas oil, whereby a sufficient quantity may be introduced into the cooling oil circulating system to initially establish circulation of this condens- 45 ing medium through the heat exchange section E of the column and the cooler 47. Surge tank T may have sufficient gas oil storage capacity to provide for thus establishing operating conditions after "shut-downs".

Variations in the quantities of feed, other factors being constant, are taken care of by variations in the quantity of bottoms produced which are automatically withdrawn through float controlled valve 36 as controlled by float control 38. 55

An example of operation is as follows: A topped Santa Fe Springs residuum having a viscosity of 348 sec. at 122° F. is introduced into the heater H at a preheated temperature of 350° F., at a rate of 1804 barrels per 24 hours. 60 The feed, after passing through the heater, is introduced into the flash section of the fractionating column at 28 at a temperature of approximately 715° F. Unvaporized oil is withdrawn from the flash tray at 30 and recirculated 65through the radiant tubes of the heater and reintroduced into the fractionating column with the feed at a rate of approximately 3500 barrels per 24 hours. Approximately 3750 barrels per 24 hours of gas oil at 115° F. is recycled to the 70 heat exchange trays and approximately 604 barrels per 24 hours of reflux returned to the top of the fractionating section. The temperature of the coolest heat exchange tray is thus maintained at approximately 200° F. and the steam

and fixed gases are exhausted from the top of the column at approximately this same temperature and at a pressure of 29.1 in. vacuum. Approximately 2 gallons per hour of caustic solution is introduced into the heated feed to the fractionating system. Under these conditions 255 barrels of gas oil having a viscosity of 145 sec. at 100° F., 529 barrels per 24 hours of lubricating oil distillate having a viscosity of 244 sec. 10 at 130° F., 322 barrels per 24 hours having a viscosity of 124 sec. at 210° F., 151 barrels per 24 hours of lubricating oil distillate having a viscosity of 355 sec. at 210° F. and 537 barrels per 24 hours of bottoms from the stripping section 15 of the fractionating column are produced.

The foregoing described process and apparatus is merely illustrative and the invention is not limited thereby, but may include any process and apparatus which accomplishes the same within 20 the scope of the invention.

We claim:

1. In a process for distilling oil, the steps comprising heating the oil in a heating zone, withdrawing the heated oil from the heating zone and 25 then commingling caustic alkali solution with the said heated oil, introducing the heated oil together with the said commingled caustic alkali solution into a separating zone where vaporiza-

tion takes place and vapors and unvaporized oil are separated, withdrawing at least a portion of the unvaporized oil containing caustic alkali from said separating zone and commingling said withdrawn oil containing caustic alkali with oil being 5 heated in said heating zone.

2. In a process for distilling oil, the steps comprising heating the oil in a heating zone, withdrawing the heated oil from the heating zone and then commingling caustic alkali solution with 10 the said heated oil, introducing the heated oil together with the said commingled caustic alkali solution into a separating zone where vaporization takes place and vapors and unvaporized oil are separated, withdrawing at least a portion of 15 the unvaporized oil containing caustic alkali from said separating zone and introducing a portion of said withdrawn oil into an intermediate section of the heating zone.

3. A process according to claim 1 in which the 20 introduced caustic solution is flashed in the center portion of the heated oil stream withdrawn from the heating zone whereby a minimum of contact of caustic solution vapors with the conduit leading to the separating zone is effected. 25

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