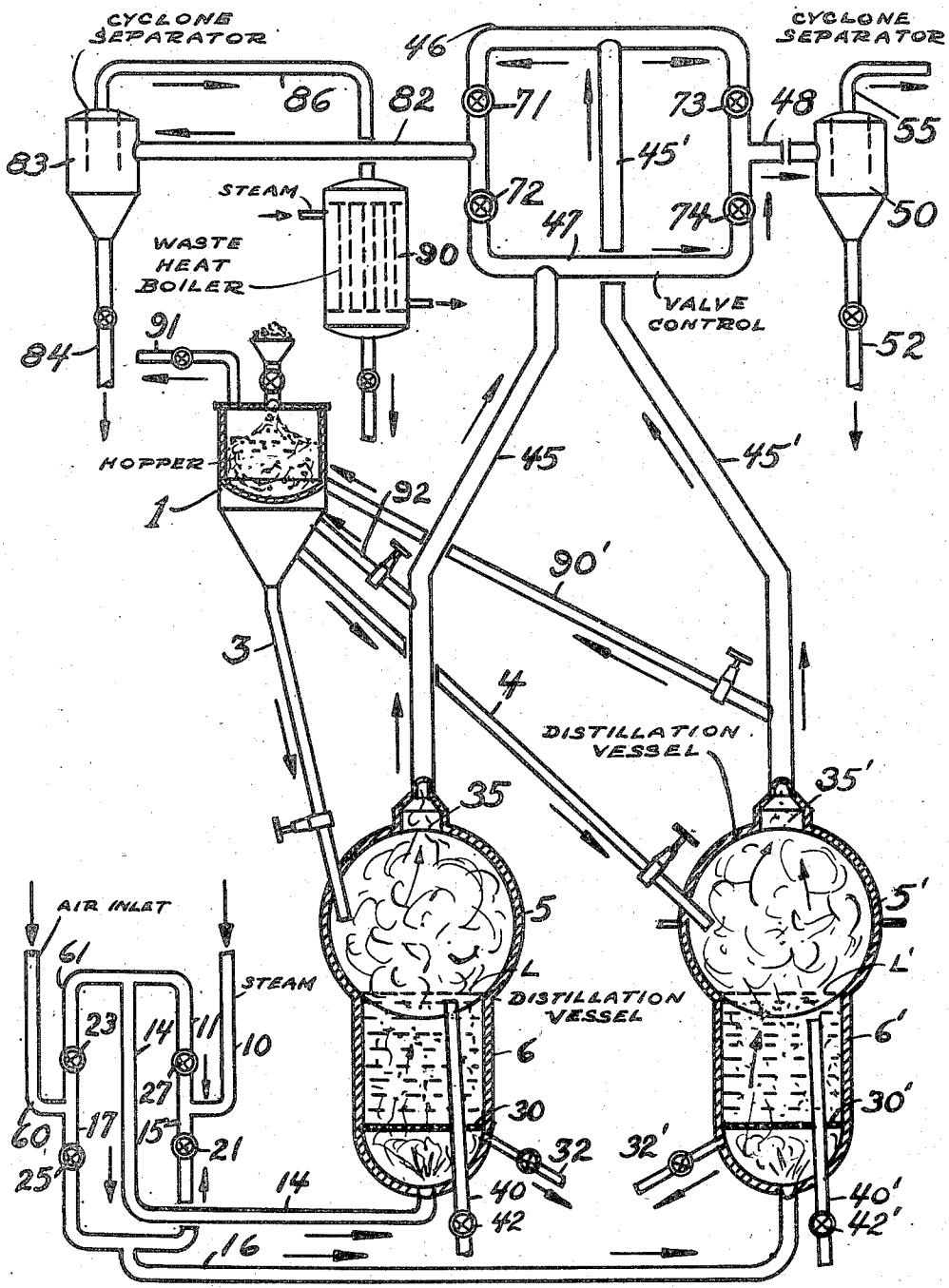
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DISTILLATION OF OIL SHALE UNDER FLUIDIZED CONDITIONS

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DISTILLATION OF OIL SHALE UNDER FLUIDIZED CONDITIONS

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2 Claims. (Cl. 202—14)

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The present invention relates to an improved method for recovering hydrocarbon oils from oil shales and other bituminous materials. More particularly, my present invention relates to a pyrolytic treatment of shale to recover therefrom maximum quantities of hydrocarbons in an economical process which may be operated continuously and in which operation the shale is subjected to a high degree of heat in the form of relatively large aggregates, thus obviating the necessity of fine grinding of the shale; furthermore, I operate the process in such a manner that the pyrolysis is effected rapidly, so that valuable hydrocarbon oil released during pyrolysis of the shale is not exposed to high temperatures in the reaction zone for too long a time and hence deterioration and degradation of said products are avoided.

According to my present invention, I have provided means for causing the shale to undergo distillation rapidly to produce valuable hydrocarbon oils, such as oils boiling within the gasoline motor fuel range, and in brief compass, my improvements in oil shale distillation involve subjecting the shale to pyrolysis in the form of a roughly ground fluidized mass admixed with hot spent shale, which latter material provides heat useful in the distillation of the fresh shale. In my operation I preferably employ two, or a multiple of two, distillation vessels to maintain continuity of operation so that while one vessel is undergoing shale distillation, the other may undergo superheating by combustion of the fixed carbon and other combustibles of spent shale. By thus operating the process in what might be termed a "blow and run" type of manipulation, I maintain continuity of the overall process, as indicated, and I also supply during the combustion operation of the spent shale substantially sufficient heat to maintain the process during the distillation operation. If desired, however, I may inject a fuel, such as liquid hydrocarbon oil, into the spent shale just prior or during the combustion thereof, if more heat is required than that obtained by burning off the fixed carbon on the spent shale.

It is therefore an object of my present invention to perform shale pyrolysis rapidly and under conditions where excessive deterioration of the product is avoided.

A further object of my invention is to reduce raw shale to aggregates of appreciable size, say of about nut coal size, and to subject the ground shale to pyrolysis.

Other and further objects of my present invention will appear from the following more detailed description and claims.

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Referring in detail to the drawing, a rough ground shale having an average size of say ¼ inch in diameter is introduced into a hopper 1 and thence discharged into a distillation vessel 5 where it mixes with a quantity of hot spent shale which is maintained in a fluidized mass in the lower portion 6 of said distillation vessel 5. The mass is maintained in the fluidized condition by discharging a gasiform material such as steam from some suitable source thru a pipe 10, thence thru a pipe 11, thence thru a pipe 14 into the bottom portion of the lower compartment 6 of distillation vessel 5. Obviously during this particular flow of steam from line 10 into vessel 5, valves 21, 23 and 25 are closed and valve 27 is in open position. Located near the bottom of vessel 5 is a grid plate 30 thru which the incoming steam passes upwardly and by maintaining the linear velocity of the up-flowing steam in compartment 6 of vessel 5 within the range of from 2–8 ft. per second with the particle size specified, a sort of dense phase suspension is maintained within the said compartment 6, that is to say, each cubic foot of steam and gas released during the distillation contains from 8–40 lbs. of shale per cubic foot of total gas and vapors. Therefore, the condition prevailing within the distillation vessel 5 is that from the grid plate 30 to a substantial level corresponding to L there is a dense phase suspension or fluidized mass of shale of varying size, the distillation causing some of the shale to disintegrate in situ to smaller aggregates, apparently by loss of adhesive material. Above L there is a solid-disengaging space where the greater internal diameter of the distillation vessel in this region causes the gas and vapor velocity to slow down so that the main bulk, at least, of the shale remains in the compartment 6, so that the vapors eventually exiting thru 35 are substantially free of entrained powdered material, or at most, contain small amounts of shale. It should be pointed out that the suspension residing in the compartment 6 is not the ordinary type of suspension in which a solid is supported in a gas, say a flowing stream of air, under such conditions that the solid material suspended in the air is swept along with the current of moving air and has no directional motion independent of the directional motion of the air stream. Rather the suspension in compartment 6 resembles a boiling liquid with some particles moving from the grid toward L and others descending from L toward the grid. This particular sort of suspension affords very

intimate and rapid mixing so that shale discharged from hopper 5 into compartment 6 is almost instantaneously mixed with spent shale or partly spent shale in compartment 6 prior to the introduction of fresh raw shale.

It will be noted that a dip pipe 40 extending into compartment 6 is provided, and this pipe 40 carries a valve 42. Normally, during the distillation operation valve 42 is open so that shale may be withdrawn thru said pipe 40 in amounts corresponding to that added thru standpipe 3 so that a considerable amount of shale may be maintained within the reaction vessel 5. However, large lumps of spent shale which collect in the vicinity of grids 30 and 30' are removed thru draw-offs 32 and 32' respectively. The steam and products of distillation are withdrawn overhead thru line 45 and thence discharged by line 47 and line 48 into a cyclone separator 50 where the last traces of shale may be removed from the vapors rejected from the system thru draw-off pipe 52. The gases and vapors, on the other hand, are withdrawn from the cyclone separator thru line 55 and delivered to fractionating and distillation equipment to recover desired products. The fractionating and recovering of desired products may be carried out in a conventional manner and is not shown in the drawing.

Referring again to reaction vessel 5, and particularly with respect to the conditions maintained therein, it is pointed out that a temperature of about 1000° F. is maintained therein. This temperature is maintained by preheating the shale entering the distillation vessel 6 to a temperature of about 400° F.–500° F. in a manner which will be subsequently explained, and causing the spent shale and the shale added to distillation vessel 5 to be so proportioned that there is present during the distillation about 2 lbs. of spent shale per pound of raw shale, the temperature of the spent shale immediately following the completion of the burning operation being within the range of about 1000° F.–1300° F. By adding preheated raw shale to this spent shale in the proportions indicated, the mixture will have a temperature of about 1000° F. and this temperature gives good results. It is pointed out, however, that good results are also obtained by using a distillation range of from 800° F.–1200° F. and by properly adjusting conditions within the reaction zone.

Employing an Australian shale, each ton of said shale will normally produce about 30 gallons of oil, and I have found that good results are obtainable by permitting the shale undergoing distillation to remain in the distillation zone for a period of 15 seconds to one minute.

During the operation described there normally comes a time when due to temperature drops, the operation in vessel 5 must be discontinued and this is accomplished by closing the valve in line 3 and, at the same time, closing valve 42 in line 40. A minute or so later, or after permitting sufficient time to complete the distillation and to remove recoverable hydrocarbons, air is admitted from line 60 to line 61 by opening valve 23. This air flows with the steam from line 11 into line 14 and thence into the bottom of the reaction vessel 5. Shortly thereafter valve 27 is closed so that only air is discharged into the bottom of vessel 5. This air, which may be at ordinary atmospheric temperature as its enters thru line 60, causes a combustion of the combustibles remaining in the spent shale which are carbonaceous in nature, and the combustion products flow upwardly thru line

45. During the discharge of hydrocarbon vapors from line 45 into cyclone separator 50, all valves except valve 74 are closed, the latter valve, of course, being open. When, however, flue gas is discharged from line 45 into line 47, valve 74 is closed, valve 71 is closed, and valve 72 is in open position so that the flue gas passes into line 82 and then passes into cyclone separator 83 where any entrained shale is separated and withdrawn from the system thru line 84, the flue gases leaving or exiting from the separator thru line 86 and passing thru a waste heat boiler 90 where a portion of their sensible heat may be recovered for use in some part of the system or for some extraneous purpose. However, a portion of the flue gas in line 45 may be discharged thru valved line 92 into the bottom of the hopper 1 where it serves to dry and preheat the shale therein, the gases then existing thru a valved line 91.

During the combustion of the spent shale in vessel 5, shale is continuously discharged from hopper 1 thru line 4 into a second distillation vessel 5' which may be identical in all respects with vessel 5, and where in order to maintain continuity of operation of the overall process, the distillation may be carried out in vessel 5' while the shale in 5 is undergoing burning.

In the drawing, I have shown the corresponding parts of vessel 5' and have indicated them by employing the same reference characters carrying a prime mark. It is believed unnecessary to repeat the operation carried out in vessel 5' because it is substantially identical in all particulars to that previously described as carried out in vessel 5. In supplying steam to vessel 6', however, it should be pointed out that steam entering thru line 10 passes thru line 15 and thence thru a line 16 into the bottom of the reactor. This flow is accomplished by opening valve 21 and closing valves 23, 25 and 27. At the conclusion of the distillation operation in vessel 6', valve 25 is opened to permit air to flow thru line 17 into line 16 and thence into the bottom of vessel 6' and shortly thereafter valve 27 is closed so that only air passes thru line 16 into the bottom of vessel 6'.

With respect to the distillation products resulting from the distillation operation in distillation vessel 5', the same are withdrawn thru line 45' and discharged into cyclone separator 50 by opening valve 73, valves 71 and 74 being in closed positions. When the gas in line 45' is a flue gas, valve 73 is closed and valve 71 is opened, valve 72 being closed so that the flue gas flows thru line 46 into line 82 and eventually into the cyclone separator 83.

As previously indicated, in order to maintain continuity of operation in a process of the type herein described, two or more reactors are required so that while one or more are undergoing burning of the spent shale, the other may be employed in the productive phase. It is possible, according to the operation which I have shown, to provide sufficient heat within the process itself to support the distillation of the shale, although in some instances it may be necessary to burn a portion of the products of the distillation, such as the normally gaseous products, in order to supplement the heat produced by the combustion of the fixed carbon on the spent shale in order to supply sufficient heat for the distillation operation.

In the prior description, I have referred to the operation of producing oil from shale as distillation. I do not wish to be bound by any theory of what actually takes place during the production of the oil in the process indicated, as it may be

that more than simple distillation is involved. In the appended claims I have referred to the operation as being pyrolytic, and by that term I mean to imply that the operation may be simple distillation, destructive distillation, metathetical or effected in some other manner caused by the application of heat.

Many modifications of my invention will readily suggest themselves to those who are familiar with this art.

What I claim is:

1. A continuous method of distilling shale to recover valuable hydrocarbons therefrom, which comprises establishing a fluidized mass of finely divided spent shale to which has been imparted a heat content adequate for distillation of raw shale, maintaining the mass in fluidized condition by discharging into it a gasiform material, feeding into said fluidized mass finely divided raw shale for rapid, intimate admixture therewith and distillation primarily by the heat already imparted to the spent shale, withdrawing spent shale in amount corresponding in general to the amount of raw shale added, withdrawing steam and products of distillation derived from the raw shale by the distillation effect of the hot spent shale, diverting the feed of raw shale to another hot fluidized mass of spent shale when the heat content of the fluidized mass of spent shale being used for distillation has dropped to a point where it is no longer effective for such purpose, passing an oxygen-containing gas into the mass of spent shale from which the feed of raw shale has been diverted, whereby the fluidized state is maintained and combustion of residual carbonaceous components of the spent shale is caused to take place,

withdrawing products of combustion, and again feeding raw shale into the fluidized mass of spent shale when it has reached the proper temperature for further use in distillation, the finely divided raw shale to be distilled being preheated by contact with hot gases from combustion of the spent shale, generated from the spent shale occupying its original place, said hot gases being passed into the finely divided raw shale so as to pass upwardly therethrough.

2. Process according to claim 1, in which the fluidized mass undergoing distillation is a dense phase suspension in which each cubic foot of gasiform material therein contains from 8 to 40 pounds of shale, and in which hot spent shale at a temperature of 1000° to 1300° F. and raw shale added to the distillation vessel are so proportioned that there is present during the distillation about 2 pounds of spent shale per pound of raw shale.

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