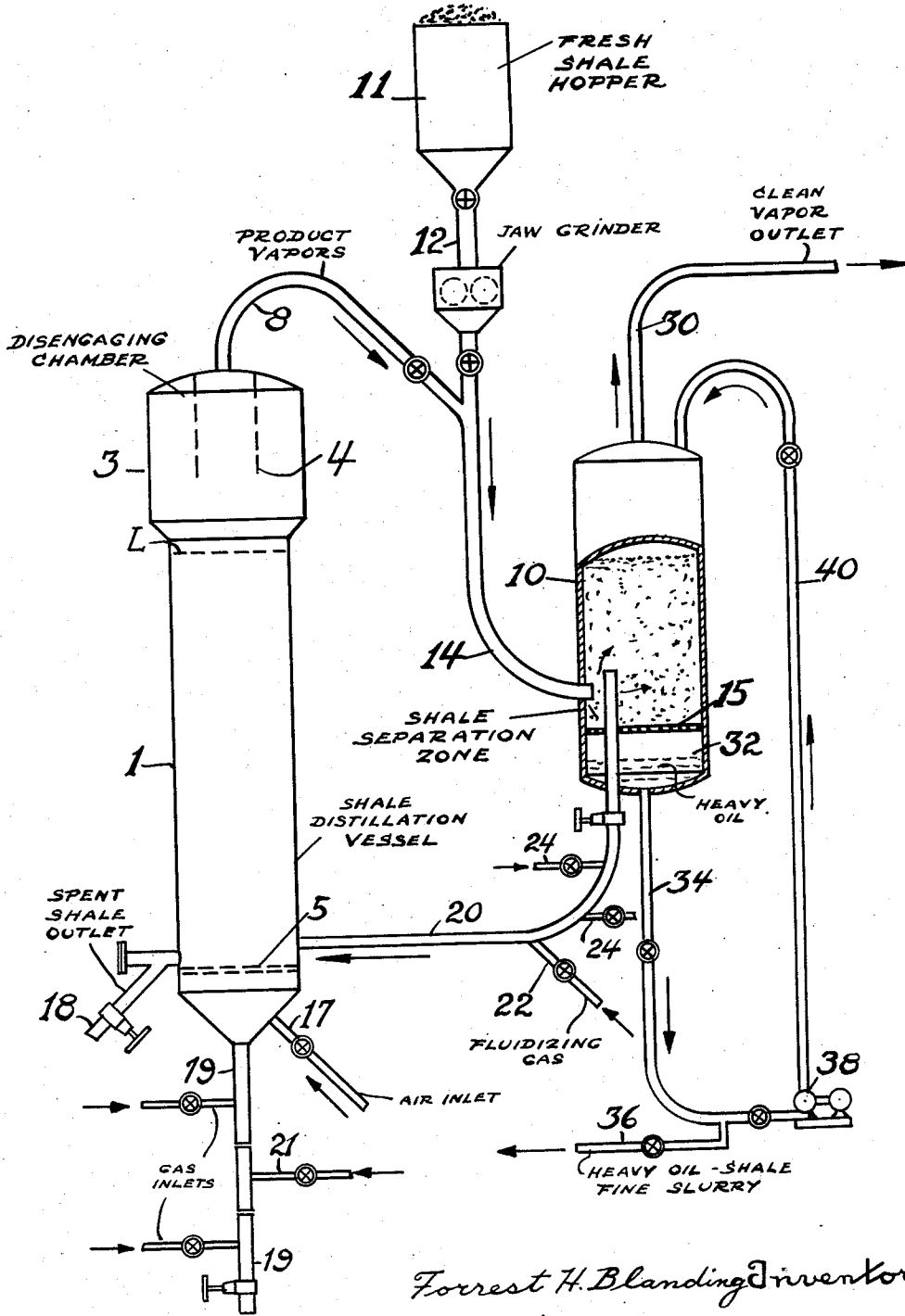


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SHALE DISTILLATION
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SHALE DISTILLATION

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2 Claims. (Cl. 202—6)

The present invention relates to improvements in the art of distilling oil-bearing materials, such as shales, oil sands, and the like, and more particularly it relates to a continuous method of distilling shale and the like in a fluidized state under conditions such that fresh shale feed in the form of lumps or aggregates less than 2 inches in diameter is charged to a vessel equipped with a grid where the shale is fluidized by means of the vapors from the shale distillation, whereupon the shale is heated to a point of incipient distillation while the vapors are cooled to a point where a portion of the heavy oil condenses out and serves to strip the vapor stream of all suspended finely divided spent shale, all of which will more fully appear hereinafter.

Heretofore it was the practice to distill shale in a stationary bed type of operation to recover hydrocarbon oil therefrom. More recently, processes have been developed for distilling shale continuously, the shale being in the form of a fluidized mass, that is to say, admixed with a moving gas in a reaction zone, the linear velocity of the moving gas being such as to maintain the shale in a turbulent, ebullient condition but, nevertheless, low enough to permit the main body of the shale to remain in the reaction zone rather than to be swept overhead with the vapors and/or gases.

The main object of my invention therefore is to provide an expeditious and practical method for continuously distilling shale.

A more limited object of my invention is to provide a continuous shale distillation process in which the shale undergoes distillation in a fluidized form or condition, including means permitting recovery from the distillation process of a relatively "clean" product, that is, substantially freed of particles of dust.

A third object of my invention is to conduct the distillation of the shale under conditions such that there is a minimum of cracking of the hydrocarbon vapors released during the distillation of the shale.

In order to afford a better understanding of my invention, I have shown in the accompanying drawing diagrammatically, a flow plan which will serve to illustrate a preferred modification thereof.

Referring in detail to the drawing, 1 represents a shale distillation zone which preferably is in the form of a cylindrical vessel having an expanded upper section 3 of greater diameter than the distillation vessel proper 1. I provide at near the bottom of the distillation vessel 1, a foraminous member 5 which may be in the form

of a screen, a grid, a rotating grate, or any other means which would permit the flow of gases and/or vapors therethrough. 10 represents a shale separation and preheating zone. The function of this chamber 10 will appear more fully as the description of my invention proceeds. At this point, I shall simply state that the fresh shale flows from a hopper 11 through line 12 in the form of roughly ground lumps produced by passing the shale through a jaw grinder or the like to line 14 carrying hot distillation products, and the mixture is fed into a point below the middle of the separation zone 10, but above the foraminous member 15. The fresh shale passes through line 12 by gravity aided by the fluidizing action of the vapors. The shale in line 12 is at atmospheric temperature, but when thoroughly mixed with the hot vapors in line 14 and later with spent shale in 10, it is preheated to a temperature of from 300-600° F., then withdrawn through a draw-off pipe 20 into which is injected a fluidizing gas at near the bottom thereof through a line 22 and thence conveyed by natural flow into the distillation vessel 1. The fluidizing gas which may be steam, flue gas, CO₂, air, or the like, is preferably injected into the draw-off pipe 20 through a plurality of taps 24 along the length of the downflow arm of stand-pipe 20, this fluidizing gas serving of course to increase the fluidity of the shale and to promote its ready flowability in the manner indicated.

In order to provide the heat necessary for the distillation in reactor 1, I cause air or some other oxygen-containing gas to contact the shale in reactor 1. Secondary air, if needed, is fed through a line 17 into the bottom of the said reaction zone wherein it serves to burn carbonaceous material from the spent shale after the distillation products have been removed and thus provide the heat necessary to sustain the distillation. Of course, if sufficient air is injected through pipe 22 no air need be injected through line 17.

As to conditions in the distillation zone, first, the linear velocity of the gases within the zone is maintained within the limits of ½ to 5 ft. per second whereupon the particles or lumps of shale are maintained in the fluidized condition previously referred to, and in the upper portion of the distillation zone 1 the shale undergoes pyrolysis and/or distillation with the release of hydrocarbon vapors. The spent shale, that is the shale from which the volatile constituents have been removed, tends to settle toward the grid 5 and is withdrawn through pipe 19 carrying taps 21 through which a fluidizing gas, such as CO₂, flue

gas, etc. may be injected into pipe 19 to facilitate the flow of shale in pipe 19. Any clinker which is formed and cannot pass through grid 5 may be withdrawn through a suitable draw-off trough 18.

The temperature within the reaction zone should be maintained around 825-1000° F., and the pressure should be around atmospheric or sufficiently above to cause the flow of vapors through the subsequent system. The operation is essentially a low pressure operation. Under the conditions stated, the mass of shale from the grid 5 to a point which I have indicated as L is in a fluidized, ebullient, turbulent state of motion resembling somewhat a boiling liquid.

The main bulk of the catalyst is retained in the reactor between the grid and level L by expanding the upper portion of the reactor into a disengaging chamber 3 where, due to the increased diameter, the linear velocity of the vapors is reduced and most of the entrained shale appearing therein gravitates back into the main body of the shale. Of course, during the distillation of the shale it undergoes physical disintegration and fines are formed. Some of these fines pass through the disengaging chamber 3 into the product line 8 and then eventually appear in the separation zone 10. However, the chilling effect of the cold fresh shale tends to condense and liquefy the heavier or higher boiling constituents of the vapors charged to the separation zone 10, with the result that this formed liquid washes the vapors and separates therefrom the shale fines which are carried over from the distillation vessel. Consequently, the vapors which are withdrawn from the separation vessel 10 through line 30 are substantially freed of fines.

The operation of separation zone 10 wherein the freshly added shale serves to cool the vapors has the additional advantage that the heavier oils, say boiling in the gas oil range and above are quenched in line 14 and are therefore prevented from undergoing as much cracking as would be obtained in conventional design. This is advantageous because of low pressure cracking were to take place, the cracked products would have a very low octane rating and be highly olefinic in nature since they would be cracked under low pressure thermal conditions. Therefore, instead of being cracked, the higher boiling vapors are quenched and at least a part of the vapors are liquefied and collected in the bottom of the separation chamber 32 and withdrawn through lines 34 and 36. This heavy oil-shale fines slurry is then sent to a classifier or settling zone (not shown) to separate the fines, and thereafter the heavy oil may be processed as by catalytic cracking to produce high quality products.

A portion of the oil in line 34 may be recycled by pump 38 in line 40 for scrubbing of the vapors to remove fines therefrom and/or further to quench the reaction products below cracking temperatures.

As I have indicated in the drawing, shale separation zone 10 need not be as large as distillation vessel 1 and the vapor and/or vapor-gas velocity therein may be up to 10 ft. per second or more. The shale which enters the shale separation zone may have a size averaging 1/4 inch in diameter, and at 10 ft. per second velocity there is very little tendency for the shale to be carried overhead.

Another important feature is that I have found by test that the behavior of a fluidized solid in a reaction chamber is not impaired by counterflow of a liquid and the fluidizing gas, such as the oil fed say through line 40 against the upflow-

ing fluidized gas. Hence I have discovered that I may cause a liquid oil to flow countercurrent to gas through a fluidized mass of solids without interfering with the state or condition of the fluidized mass, i. e., while maintaining the solids in fluid condition.

Various expedients may be resorted to, to improve my process. For instance, I prefer to dispose solid separator 4 in disengaging chamber 3 in order to separate at least a portion of the fines. However, at high temperatures "cyclone separators," so-called, are not too efficient and neither are electrical precipitators. Experience has shown that they are apt to operate poorly. Hence I do not rely on these types of separators to remove the fines completely from the product vapors, but as indicated I may use them to separate such fines as they are enabled to accomplish and to employ the technique described in connection with the description of the operation of separation zone 10.

It will be noted that in the system I have shown, I have not included conventional tubular heat exchangers. I have purposely omitted these for it has been shown that residual shale ash fines in the product vapors deposit out in cooling coils and cause serious plugging. Hence, the ordinary heat exchanger is not satisfactory. On the other hand, it has been demonstrated by experimental data that substantial improvements in oil yield reduction of the air requirement to the process, and substantial increase of throughput to a given unit could be obtained if the shale were preheated. Hence, it is advisable to preheat the shale and I preheat it in the manner indicated. I preheat it by contact with the hot product vapors containing an appreciable amount of shale ash fines which pass through pipe 8 at a velocity of 5-30 ft. per second. Almost immediately in this pipe and without the use of other heat exchanger equipment, the shale fed to the reaction zone is heated to about 300-600° F. or the maximum temperature to which it can be heated without appreciable cracking of the oil contained therein.

In order to show the utility of my invention, I shall describe at this point the processing of an Australian oil shale. This shale yielded 2 1/2 barrels of fuel oil per ton of shale, when operating in reactor 1 at a temperature of 950° F., a pressure of 8 lbs. per square inch gauge, and a gas velocity of 3 ft. per second. Air was admitted for burning at a temperature of 350° F. In addition to the fuel oil, I also obtained 0.16 barrel of light naphtha per ton of shale and about 6 weight per cent of C₃ and lighter hydrocarbons based on the oil shale fed.

I set forth below a table which shows the effect of temperature on yields, and from the table it will be noted that the higher temperatures produce less oil, but I have found that the fuel oil quality is better at the higher temperatures and consequently the use of higher temperatures has advantages:

	Temperature		
	850° F.	900° F.	1000° F.
Fuel oil..... bbl./ton.....	2.60	2.50	2.15
Naphtha..... do.....	.12	.16	.27
Dry gas--weight per cent on shale.....	4	6	15.5

I have not indicated the manner of processing the vapors in line 30. These gasoline and other hydrocarbon vapors may be condensed and col-

lected and otherwise treated to produce a commercial product in known manner.

To summarize the advantages which I believe to flow from my invention, I set forth the following:

(1) The problem of separating the spent shale fines from most of the product vapors is accomplished without the necessity for other equipment. It might, however, be desirable to include one centrifugal separator in line 14 to remove some of the solid fines.

(2) Product vapors are quickly quenched in line 14 because the necessity of removing the solid particles in the hot vapor has been eliminated. This reduces the undesirable product cracking and degradation.

(3) Heat has been externally recovered. This will decrease air requirement per pound of shale, and increase the permissible throughput to a given amount. Also, yields of oil based on shale will be increased because less carbon and hydrogen would need to be burned therefrom.

(4) Product vapors leaving the separating chamber will be scrubbed clean. Therefore, they can be passed through coolers without plugging troubles.

Numerous modifications of my invention may be made without departing from the spirit thereof.

What is claimed is:

1. A continuous method of distilling shale to produce valuable hydrocarbon oils which comprises providing a fluidized mass of the shale in a distillation zone, withdrawing hydrocarbon vapors containing shale fines from said zone, mixing sufficient fresh cold shale with said hydrocarbon vapors to quench the same and to cause condensation of the higher boiling constituents thereof, charging the mixture of vapors, condensate and cold shale to a separation zone where the shale is

maintained in a fluidized condition by the upward movement of the hydrocarbon vapors, withdrawing condensate from the separation zone and recycling a portion of said condensate to the top of said separation zone where it flows countercurrent to the vapors passing therethrough and causes shale fines to be removed from said vapors by the scrubbing action of the said condensate, recovering hydrocarbon vapors and product vapors from the separation zone, withdrawing preheated shale from the separation zone and charging it to a shale distillation zone.

2. A continuous method of distilling solid carbonaceous material to produce valuable hydrocarbon oils which comprises providing a fluidized mass of the solid carbonaceous material in a distillation zone, withdrawing hydrocarbon vapors containing solid carbonaceous material fines from said zone, mixing sufficient fresh cold solid carbonaceous material with said hydrocarbon vapors to quench the same and to cause condensation of the higher boiling constituents thereof, charging the mixture of vapors, condensate and cold solid carbonaceous material to a separation zone where the solid carbonaceous material is maintained in a fluidized condition by the upward movement of the hydrocarbon vapors, withdrawing condensate from the separation zone and recycling a portion of said condensate to the top of said separation zone where it flows countercurrent to the vapors passing therethrough and causes solid carbonaceous material fines to be removed from said vapors by the scrubbing action of the said condensate, recovering hydrocarbon vapors and product vapors from the separation zone, withdrawing preheated solid carbonaceous material from the separation zone and charging it to a solid carbonaceous material distillation zone.

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