

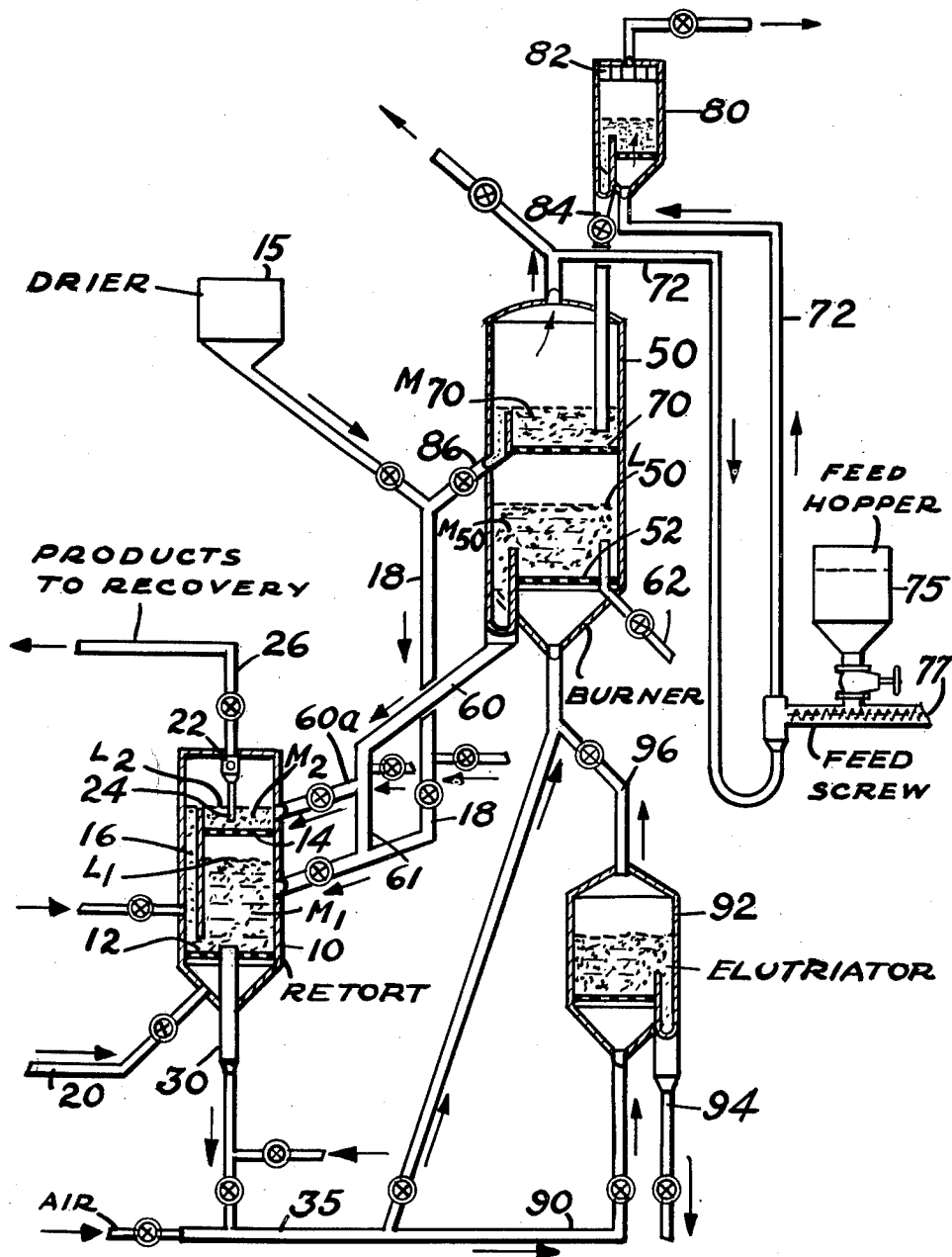
Nov. 18, 1952

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2,618,588

FLUIDIZED SHALE DISTILLATION

Filed June 21, 1949



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UNITED STATES PATENT OFFICE

2,618,588

FLUIDIZED SHALE DISTILLATION

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Application June 21, 1949, Serial No. 100,327

4 Claims. (Cl. 202-14)

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The present invention relates to the art of distilling oil-bearing minerals such as oil shale, oil sands, tar sands and the like, maintained in the form of subdivided particles in a highly turbulent state, fluidized by upwardly flowing gases to resemble a boiling liquid, wherein the fresh charge undergoes rapid disintegration to form an excessive amount of particles having sizes below a properly fluidized range. More particularly, the invention relates to improved means for maintaining properly fluidized beds of such materials during distillation.

Prior to the present invention, it has been proposed to carry out the distillation of oil shale in the form of subdivided solids varying in particle size from a fine powder up to rather large aggregates of, say, about $\frac{1}{4}$ in. diameter in a highly turbulent fluidized state. The heat required for distillation has been supplied either as sensible heat of preheated process materials, by indirect heat exchange of the fluidized bed with suitable heating means, by combustion of combustible shale constituents within the retorting zone, or as sensible heat of hot spent shale highly heated in a separate combustion zone and circulated to the fluidized bed in the retorting zone. The principal problem encountered in all these operations arises as the result of the strong tendency of the shale to disintegrate rapidly in the course of the distillation to particles having an extremely small size of about 0-20 microns which is the unit particle size of the shale silt.

For example, Colorado shale when subjected to a fluid-type distillation quickly forms a mass containing about 60 per cent of fines of 0-20 microns size, and this even if the shale is charged in rather coarse aggregates. Attrition further increases the proportion of these fines in the highly turbulent or circulating solids masses. At the condition of fluid shale distillation such fines tend to be entrained and carried overhead by the fluidizing gases. This situation is further aggravated by relatively frequent localized spurts or jets of gasiform materials caused in the fluidized bed by the flashing of water and oil from the cold fresh shale dropped into the hot fluidized bed. Even gas-solids separators of normally highest efficiency have been found inadequate to cope with this degree of solids entrainment. Aside from severe losses of valuable carbonaceous constituents removed from the distillation zone with these fines, the carry-over of excessive amounts of fines seriously complicates liquid product recovery due to heavy slurry formation and detrimentally affects the fluidity of the fluidized bed which may be rapidly converted into a dilute

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phase which cannot be retained in the distillation zone unless special precautions are taken. The present invention provides means for effectively counter-acting the detrimental effects of this type of fines entrainment.

In accordance with the present invention, in its broadest aspect, losses of shale fines having particle sizes of 0-20 microns from a fluidized shale bed undergoing distillation, by entrainment and carry-over of the fines in the fluidizing gases and gasiform distillation products, may be materially reduced by obstructing the upward flow of the gases and entrained solids above the interface of the fluidized bed and within the distillation zone in such a manner that gasiform materials may pass the obstruction completely while a proportion of the entrained solids is deflected back into the fluidized bed. This obstruction may have the form of inclined or horizontal offset baffles prescribing a zig-zag flow for the solids-in-gas suspension, or of a non-fluidizable stationary packing having irregular passageways, or the like. However, in accordance with a preferred embodiment of the invention, the obstruction has the form of a horizontal perforated plate or grid capable of supporting a second fluidized solids mass above that undergoing distillation.

All types of obstructions mentioned afford the advantage of returning a proportion of entrained fines by deflection continuously to the shale bed undergoing distillation and thus of reducing fines carry-over into the product recovery system and its detrimental effects. However, when a grid plate is used, permitting solid particles to penetrate the perforations, a second fluidized bed of particles will form above the grid within a relatively short time of operation. Such fluidized solids have a strong capacity for collecting fines of 0-20 microns size up to a concentration of about 30-50 per cent. A more specific and preferred embodiment of the invention provides, therefore, for the maintenance of a fluidized bed of solids supported by a horizontal grid plate above the interface of the fluidized shale bed undergoing distillation.

The formation of such a second fluidized bed by direct carry-over of particles from the retorting bed may be replaced or supplemented by the addition of subdivided solids by means other than immediate carry-over from the retorting bed. The preferred embodiment of the invention involves the supply to the second bed of spent shale which has been subjected to combustion in a separate combustion zone for the purposes of generating the heat required for distillation.

The two fluidized beds are preferably con-

ducted by a conventional downcomer pipe which permits flow of solids from the upper fluidized bed to the lower retorting bed. In this manner, a substantial proportion of the solids carried over from the retorting bed into the second bed may be returned to the retorting bed. In addition, part or all of the heat-carrying burned shale recirculated from a separate combustion zone may be passed through the upper bed to the retorting bed and thus serve the dual purpose of preventing loss of shale fines from the distillation zone and supplying heat to the retorting bed.

Having set forth its objects and general nature, the invention will be best understood from the following more detailed description wherein reference will be made to the accompanying drawing, the single figure of which is a semi-diagrammatic illustration of a system suitable to carry out a preferred embodiment of the invention.

Referring now in detail to the drawing, the numeral 10 designates a mostly conventional distillation vessel or retort adapted for fluid-type shale distillation and containing a fluidized shale bed M_1 supported by a gas distributing device, such as grid 12, and having a well defined interface L_1 . Above this interface a second perforated grid 14 is provided. An overflow downcomer 16 leads from a point above grid 14 to a point below interface L_1 .

In operation, fresh coarse shale, which may have a particle size of about 4-50 mesh, may be supplied from feed hopper 15 through line 18 into the space between interface L_1 and grid 12. A fluidizing gas, such as steam, product tail gas, other inert gas, CO_2 and/or air, is supplied through line 20 and grid 12 at such a rate that a linear superficial gas velocity of about 0.5-3 ft. per second, suitable for fluidization, is established within bed M_1 . Bed M_1 may be maintained at a distillation temperature of about 850°-1100° F. by any of the conventional methods described above.

The fresh shale upon entering bed M_1 , undergoes distillation and disintegrates rapidly. As a result, bed M_1 may have a particle size distribution about as follows:

Size:	Weight per cent
0-20 microns.....	55
20-80 microns.....	10
80 microns, -35 mesh.....	15
On 35 mesh.....	20

A portion of the entrained solids impinges on the impermeate portions of grid 14 and is thus returned to bed M_1 . The remainder of the entrained solids penetrates the perforations of grid 14. Initially the fines of 0-20 microns size carried through grid 14 may remain entrained in the gases and vapors and may be carried therein to a gas-solids separator 22 in which a substantial proportion of the entrained fines, say, about 70-90%, may be separated. The separated fines may be returned to bed M_2 via dip-pipe 24, gradually building up thereon until a second fluidized bed M_2 having an interface L_2 is formed above grid 14. Thereafter, a major proportion of the fines penetrating grid 14 is retained in bed M_2 , the remainder being carried into separator 22. Equilibrium of the system is established and maintained by overflowing and returning solids from bed M_2 via downcomer 16 to bed M_1 and withdrawing spent shale from bed M_1 through line 30. In this manner, about 80-99% of the solids leaving bed M_1 entrained in gases and vapors will be returned to bed M_1 via downcomer 16.

As previously indicated, the invention has particular advantages when used in connection with a system wherein hot spent shale is circulated from a separate combustion zone to the retorting zone for the purposes of heat supply. A system specifically adapted to the application of the invention is illustrated in the drawing.

As shown in the drawing, a fluidized combustion zone or burner 50 is provided in addition to the elements just described. Spent shale withdrawn through line 30 from bed M_1 may be passed to line 35 and suspended therein in air sufficiently preheated to support combustion of the shale. The air is supplied at a rate sufficient to carry the spent shale through grid 52 into burner 50, to burn part or all of the carbon off the spent shale at a temperature about 50°-300° F. higher than that in retort 10, and to maintain a superficial gas velocity within burner 50 of about 1-3 ft. per second, adequate to establish a fluidized mass M_{50} of burning shale having an interface L_{50} within burner 50. The gas containing entrained shale fines passes overhead from level L_{50} and may be further handled in any desired manner.

Burned spent shale is withdrawn from bed M_{50} through line 60 which may be an aerated stand-pipe or the like and passed substantially at the temperature of mass M_{50} to bed M_2 in retort 10. The circulation rate of hot shale through line 50 depends on the temperature differential between burner 50 and bed M_1 , and upon the heat requirement in vessel 10. A ratio of burned, hot shale to fresh shale charged of about 5-20:1 is usually adequate for the purposes of heat supply. Approximately the same solids circulation rate is maintained in lines 30 and 35 to establish equilibrium. Excess solids may be withdrawn from burner 50 through line 62.

The particle size distribution of the solids circulating through line 60 may be about the same as that specified above for bed M_1 .

In addition to containing material coarser than 20 microns, these particles are relatively low in carbon or practically carbon-free and have, therefore, a substantially lower gas buoyancy than the high carbon particles of similar size which are entrained and carried overhead from bed M_1 . The burned shale particles supplied through line 60 to retort 10 above grid 14 are, therefore, ideally suited to form a well fluidized, relatively dense bed of solids, M_2 , above grid 14, adapted to retain substantial proportions of fines entrained from bed M_1 and penetrating grid 14. The hot solids then flow through downcomer 16 to supply the heat required in retorting bed M_1 and to repeat the cycle described above. As a result of the high circulation rate of hot low-carbon solids from burner 50, bed M_2 will at all times consist predominantly of particles of sufficiently low gas buoyancy to retain an adequate proportion of the fines of 0-20 microns size entering bed M_2 from bed M_1 . When operating in the manner last described, the fines concentration of the effluent of retort 10 leaving through line 26 will be substantially less than in the absence of a second bed M_2 .

As an alternative to the procedure described above, the system shown in the drawing may be operated as follows.

Heater 50 may be divided by a second grid 70 into two sections with the burning taking place in the lower section. The hot flue gases from this lower section then pass up through grid 70 into a second dense phase M_{70} . The gases are cooled in this section, withdrawn through line 72 and

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used to supply the fresh shale to the system as follows.

Feed hopper 15 is replaced by hopper 75 from which the fresh charge may be supplied to line 72 by any suitable means, such as a screw conveyor 77. The gases in line 72 are then used for conveying the shale feed to an elevated feed hopper 80. Separators 82 may be located in the top of elevated feed hopper 80 to remove entrained solids. The shale from hopper 80 then flows through line 84 to bed M₇₀ wherein it is preheated by the flue gases penetrating grid 70. The preheated shale may thereafter be fed via lines 86 and 18 to bed M₁ in retort 10.

The advantages of this type of flow are as follows. The flue gases are cooled immediately so that piping and equipment following the heater are at a low temperature. This avoids expansion problems and expensive materials of construction. The feed shale is preheated by the flue gases so that less burning is required and less air is used. The flue gases are under sufficient pressure to be used in conveying the feed shale to the elevated feed hopper. This eliminates the necessity of compressing recycle gas for this purpose. A hindered settler is used as heat exchanger, thus eliminating expensive exchanger surface.

The system illustrated in the drawing permits of various modifications. Bed M₂ may serve as a cracking zone for the distillation products liberated in bed M₁. Cracking may be controlled by the amount of hot burned shale introduced through line 60. For this purpose, a portion of the hot shale in line 60 may be fed directly to bed M₁ through lines 61 and 18. Instead of passing all of the spent shale from line 30 through line 35 directly to burner 50, any desired portion of the suspension of spent shale in air may be passed from line 35 through line 90 to an elutriator 92 where shale of undesired particle size may be removed through line 94, the remainder being passed through line 96 to burner 50 to be further treated as above described.

While most of the volatile components present in the feed shale will be recovered by retorting in vessel 10, it has been found that further quantities of valuable products often can be obtained by further heating to a higher temperature. For example, ammonia and light fuel gases can be recovered by heating the spent shale from vessel 10 or vessel 50 to temperatures of the order of 1200°-1800° F. Such heating may be accomplished using indirect heating, or by recirculation of heated solids from a heater vessel, or by direct injection of an oxygen containing gas.

In many cases the spent shale rejected from the retorting system will still contain appreciable amounts of combustible material. These spent solids can be used as fuel, as for example in a boiler house. Moreover, they may be used for the generation of fuel gas or synthesis gas by contacting with steam, and/or carbon dioxide at suitable conditions. Heat for this process can be supplied by solids recirculation from a heater

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vessel, or by direct injection or an oxygen containing gas.

Other modifications of the systems illustrated may appear to those skilled in the art without deviating from the spirit of the invention.

The above description and exemplary operations have served to illustrate specific embodiments of the invention but are not intended to be limiting in scope.

What is claimed is:

1. In the process of distilling oil-bearing minerals of the type of oil shale, which disintegrate when subjected to distillation, in the form of a dense turbulent mass of subdivided solids fluidized by an upwardly flowing gasiform medium to form a well defined interface within a distillation zone, the improvement which comprises feeding said oil-bearing minerals to said mass, obstructing the upward flow of said medium containing entrained solids within the distillation zone at a level above said interface by an obstruction, which permits said medium to flow upwardly over the entire height of said distillation zone past said obstruction, forming a second fluidized mass of solids within said zone above said obstruction, continuously circulating retorted mineral particles from said first-named mass to a separate fluid-type combustion zone, maintaining at least two superimposed fluidized beds in said combustion zone, subjecting said retorted minerals to combustion in the lower one of said beds, passing hot flue gases from said lower bed through the upper one of said beds, supplying fresh minerals to said upper bed to be dried and preheated therein by said flue gases, and passing preheated minerals from said upper bed to said first-named fluidized mass.

2. The process of claim 1 in which volatile products of distillation are subjected to cracking in said second mass.

3. The process of claim 1 in which flue gases produced in said combustion zone are used to lift fresh minerals to an elevated point.

4. The process of claim 1 in which solid combustion residue from said lower one of said beds is returned to said second fluidized mass.

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