April 18, 1961 A. E. KELLEY 2,980,600

PROCESS AND APPARATUS FOR BITUMINOUS SAND TREATMENT

Filed July 19, 1957 2 Sheets-Sheet 1

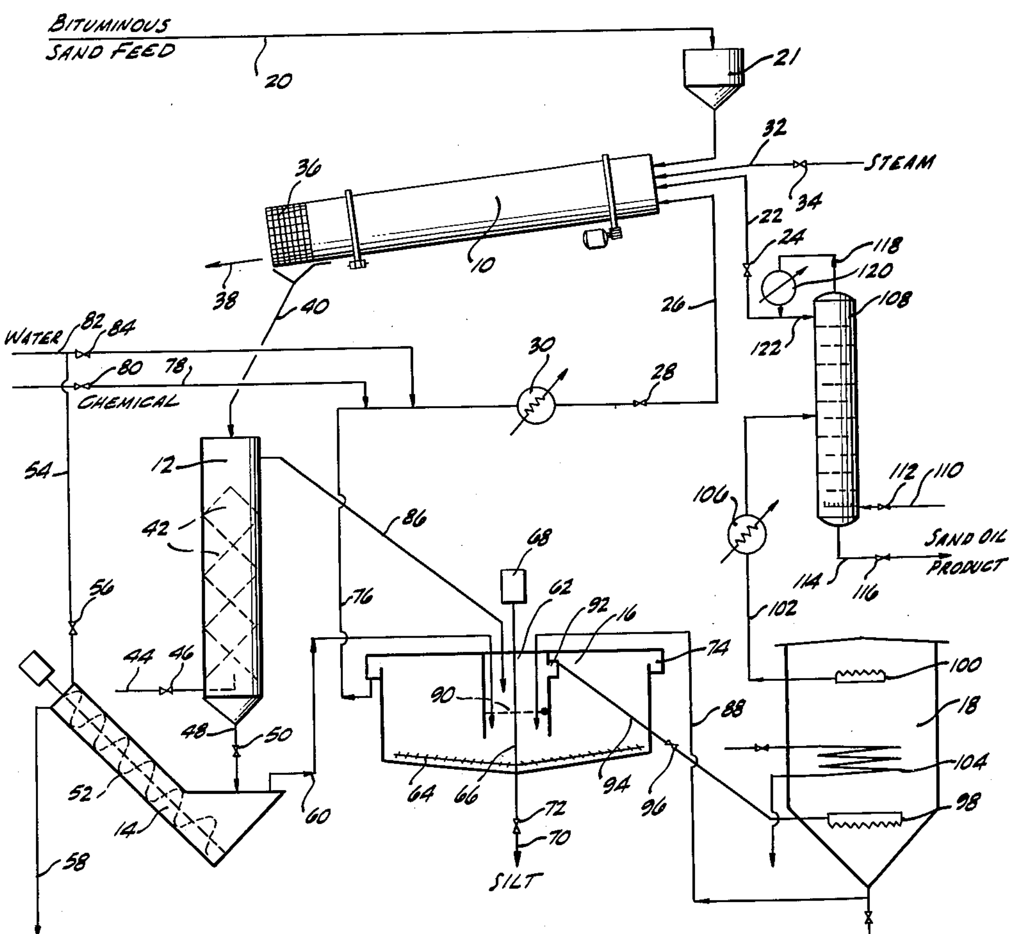

FIG. 1.

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FIG. 2.

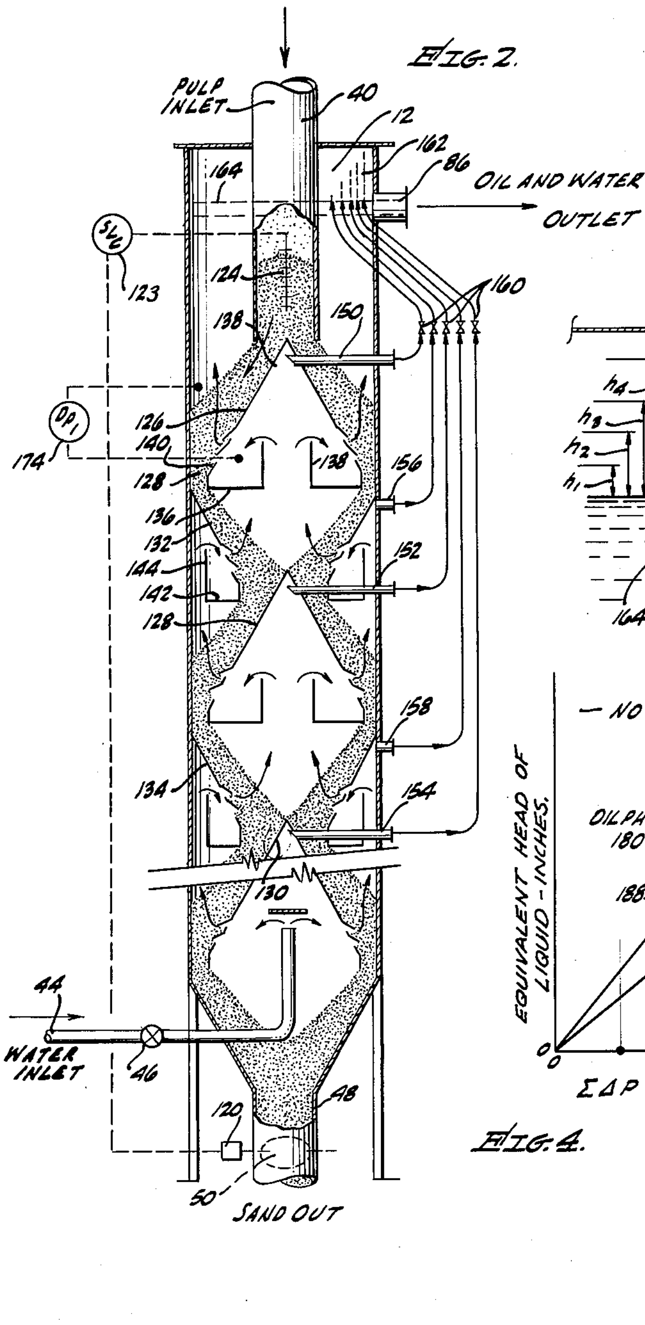
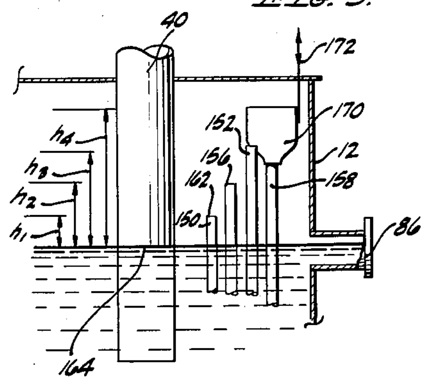


FIG. 3.



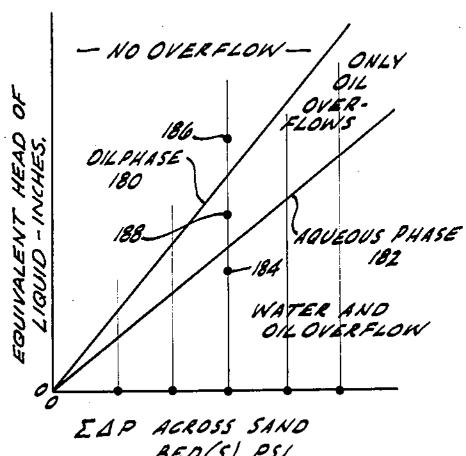


FIG. 4.

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PROCESS AND APPARATUS FOR BITUMINOUS SAND TREATMENT

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10 Claims. (Cl. 208—11)

This invention relates to the recovery of hydrocarbons from hydrocarbon-containing solids such as tar sand, oil-soaked diatomite, and the like. This invention particularly relates to an improved process and apparatus for treating such materials at relatively low temperatures utilizing particularly efficient washing and separation steps to effect a substantially complete recovery of the hydrocarbon material present.

Extensive deposits of tar sands or bituminous sands are known to exist at widely separated places in the world. These materials are essentially silicious materials, such as sands, loosely agglomerated sandstones, or diatomaceous earth, saturated with relatively heavy or viscous hydrocarbon materials resembling low gravity crude petroleum. They exist near the surface of the earth and are generally discovered through location of their outcroppings. Extensive deposits of such materials have been discovered in the Athabaska region of Northern Alberta, Canada, in the Uinta Basin near Vernal in Northeastern Utah, and in the Santa Maria area of Southern California about 130 miles northwest of Los Angeles. In this latter area extensive deposits are found in the Sisquoc River Valley, near Casmalia, and elsewhere.

Surveys of these deposits have reavealed that they contain tremendous quantities of hydrocarbon materials very similar to low gravity crude petroleum, and individual deposits have been estimated to contain on the order of 60 to 70 million barrels of tar sand oil. Extensive recovery of these oils has not been achieved, primarily because of the expense in relation to crude petroleum in spite of the fact of the accessibility of the material near the earth's surface. However with rising costs of crude petroleum due to production and depletion of known petroleum reserves, an efficient and economical process and apparatus for the treatment of such bituminous sands has become highly desirable.

The principal disadvantage in previous processes lies the extensive requirement of reagent and in the difficulty of separating the very heavy oil from the sand or other solid grains after the pulping or treating step. The present invention successfully overcomes these disadvantages through the utilization of a particularly efficient method of treating the pulped material to effect sand separation while avoiding oil rewetting.

In the following description the phrases "bituminous sand" or "tar sand" are used to refer generally to all granular solid bituminous or petroliferous materials soaked with a usually highly viscous liquid or semi-liquid hydrocarbonaceous material, although it specifically refers to a characteristic type of bituminous solid consisting of discrete particles of sand bound together by a continuous viscous hydrocarbon oil phase. This terminology is used for the sake of simplicity of description, and it should be understood that the process and apparatus herein described may be applied to other solids similarly containing a bituminous or viscous hydrocarbonaceous coating.

The present invention is directed to a low temperature process using a warm aqueous solution of a special alkali metal silicate, with or without other reagents, and a moderately heavy hydrocarbon diluent to separate the heavy oil from the bituminous sands, and in which process special procedures and apparatus are used in handling the effluent from the mixing step in which these materials are heated and agitated with one another to effect the separation of the heavy oil from the sand.

It is a primary object of this invention to provide an improved process for the separation and recovery of heavy oil from bituminous solids such as tar sand and the like.

It is a specific object of this invention to treat and recover hydrocarbon oil from tar sand by pulping it with a mixture of aqueous alkali metal silicate with or without other reagents and a hydrocarbon solvent at slightly elevated temperatures to separate the oil from the sand.

It is a particular object of this invention to provide in this process a preliminary separation step applied to the pulp flowing from a pulper or mixer to produce substantially clean sand and a mixture of the aqueous chemical and the oil phases together with the step of treating the sand to free from it all of the mechanically occluded oil.

It is a further object of this invention to provide an improved apparatus adapted to effect the foregoing objects.

Other objects and advantages of this invention will become apparent to those skilled in the art as the description and illustration thereof proceed.

Briefly, the present invention comprises, an initial step of feeding the sand in chunks continuously through a feed hopper which controls the rate of flow to a mixer. Here it is mixed and pulped with an aqueous sodium silicate solution and a hydrocarbon solvent at a slightly elevated temperature. This mixing continues for a period of between about 0.2 and about 2.0 hours and at a temperature of between about 160° F. and about 250° F. Preferably this mixer is of the rotary kiln type provided with internal baffles and conveyor flights so as to control the residence time of the material in the mixer. This treatment reduces the tar sand chunks to a heavy slurry or pulp of sand, water, and oil.

The effluent from the mixer or pulper is a slurry or pulp of treated sand, aqueous chemical solution, and a hydrocarbon phase including the separated bitumen and the relatively light diluent oil. This slurry or pulp is discharged immediately to a primary separation zone in which a very rapid separation of the treated solids is effected. This leaves a stream of fluid including the hydrocarbon and aqueous phases. Since there is a considerable quantity of sand present at all times in this processing step, it is essential that some slight sand agitation be effected in order to liberate residual oil droplets which are trapped in the downwardly progressing sand during the dropout of the sand grains from the fluid phases. The sand is discharged at the bottom of the primary separator into a washer-drier in which a considerable quantity of the water present in the sand stream is recovered for recirculation. If desired, makeup water to the process may be added at this point to recover residual silicate solution from the sand as well.

From the top of the primary separator are discharged the aqueous and hydrocrabon phases substantially free of sand grains but containing variable amounts of very fine solids such as silt and clay. In the separator thickener zone, to which these phases flow, a substantially complete removal of these silt-like solids is effected from the aqueous phase and a clean water stream is produced for recirculation. A concentrated wet oil phase is discharged therefrom into a settling zone such as a wash tank in which the material is allowed to stand for periods of between about 5 and 25 hours to produce essentially water and silt-free oil, the oil being a dilute mixture of hydrocarbon diluent and the relatively heavy hydrocarbon or bitumen separated from the sand in the process. This oil phase is at some point treated as by distillation to recover the diluent oil for recirculation to the pulper. An aqueous phase containing the silt is recirculated from the settler back to the thickener zone to produce clear water. From the thickener zone is removed a concentrated slurry of silt and water which is discharged to outdoor settling basins.

As illustrated by the following examples and as described herein, the specific steps taken in the separator and settling zones to prevent contact of the sand with separated oil and to recover mechanically trapped oil from the settling sand have been found to be extremely important in the successful recovery of up to 99.9% of these heavy oils and in the production of clean sand containing less than 0.10% of the original oil.

The process of the present invention is best described and illustrated by reference to the accompanying drawings in which:

Figure 1 is a schematic flow diagram showing portions of the apparatus in elevation view,

Figure 2 is an elevation view in cross section of the primary separation zone of this invention,

Figure 3 is an enlarged detail view of the top of the primary separator, and

Figure 4 graphically illustrates the effect of the adjustment of the height of the various oil outlet lines.

Referring now more particularly to Figure 1, the essential equipment elements employed in the process and apparatus of the present invention include pulper or mixer 10, primary separator 12, sand washer and drier 14, thickener 16, and product settler 18. The subsequent discussion of the invention in connection with Figure 1 will be conducted as a typical example of the process and apparatus of this invention applied to the treatment of Sisquoc bituminous sand at a rate of approximately 200 tons per day. Although the tar sand may contain between 20 and 40 gallons of oil per ton and have a gravity from 2 to 10° API, a typical bituminous sand contains about 30 gallons per ton of 4° API gravity bitumen.

The freshly mined bituminous sand is introduced into pulper 10 by means of conveyor 20 at a rate of 200 t./d. (tons per day) controlled by solids feeder 21. A light coker gas-oil as diluent oil is introduced at a rate of 191 b./d. (barrels per day) and a temperature of 180° F. through line 22 at a rate controlled by valve 24. Also introduced into the pulper is the aqueous alkali metal silicate solution with or without other reagent which flows through line 26 at a rate of 286 b./d. controlled by valve 28. This material is maintained at a temperature of about 180° F. by means of heater or exchanger 30. To maintain a pulper temperature of about 180° F. within pulper 10, steam at the rate of 482 pounds per hour is also introduced through line 32 at a rate controlled by valve 34.

The relative rates at which the foregoing ingredients are introduced into pulping zone 10 are specific to one typical operation. In general however they are preferably maintained within certain limits in order to effect the most rapid and efficient liberation of the bituminous material from the sand or other solid grains. Pursuant to this the diluent hydrocarbon rate is that sufficient to produce an oil phase having an API gravity above 10.0°, and is preferably maintained between limits of about 0.1 and about 2.5 b./t. (barrels per ton) of raw bitumen sand feed. The aqueous silicate solution is introduced at a rate maintained between about 0.75 and about 5.0 b./t. of raw sand feed, and preferably between about 1.0 and 1.5 b./t. This aqueous solution contains between about 0.5 and 20, and preferably between about 0.75 and about 10.0 pounds of an aqueous sodium silicate concentrate per barrel. This concentrate is a 34% by weight aqueous solution and is a special material marketed commercially under the name "Silicate 120." It has a Na_2O to SiO_2 ratio of about 0.55 mol per mol. Other high basicity sodium silicates may be substituted provided this ratio is above about 0.4 and preferably greater than about 0.5. The commercial water glass of commerce is not satisfactory since it has a ratio of about 0.25.

The pulping temperature must be maintained higher than about 160° F. and preferably is maintained above 180° F., although it ordinarily should not run above about 250° F. The operation of the pulping zone is controlled relative to the set rate and the size of the pulper so that the raw bituminous sand is subjected to the action of steam, the aqueous silicate, and the hydrocarbon diluent within the pulping zone for a period of between about 0.1 and 2.0 hours. Under the conditions given previously a pulping time of about 0.25 hour will liberate substantially all of the bitumen from the sand and produce a spent sand containing less than about 3.0 pounds of hydrocarbon per ton.

The discharge end of pulping zone 10 is provided with trash screen 36 by means of which rocks and nondisaggregated lumps of tar sand are discharged from the system by means of conveyor 38. The fluid pulp discharges through the screen 36 and flows by means of line 40 into the top of primary separation zone 12. This stream contains approximately 58 t./d. of water, 55 t./d. of oil and 172 t./d. of sand. Primary separation zone 12 operates at a temperature a few degrees below that of the pulper. This is attained by making line 40 as short as possible and providing for the immediate transfer of the pulp from the pulper into the primary separator. Preferably line 40 is an inclined pipe having a slope of not less than 60° relative to the horizontal.

The interior of primary separation zone 12 is provided with a plurality of baffles 42 over which the settling sand progresses in sequence to provide the gentle agitation necessary to liberate mechanically trapped oil drops from the sand stream. Additional agitation is provided by introducing fluid hereinafter more fully described into the bottom of primary separation zone 12 through line 44 at a rate controlled by valve 46.

From the bottom of primary separation zone 12 the treated sand discharges through line 48 at a rate controlled by valve 50, which may be a density valve responsive to the density of the sand-water slurry collecting in the bottom of primary separation zone 12. In any event, the sand discharges at a rate of 172 t./d. into washer 14 along with 193 t./d. of water. The sand is picked up and conveyed upwardly by means of conveyor 52 whereby a gravity separation of the aqueous phase is provided. Preferably, part or all of the makeup water to the system is introduced by means of line 54 controlled by valve 56 as wash water to the washer-drier. The clean, oil-free sand is discharged from washer-drier 14 by means of line 58 and is conveyed to a suitable disposal point.

The aqueous phase removed with the sand from the primary separation zone 12 is separated from washer-drier 14 through line 60 and is discharged into the central well 62 of thickening zone 16. This stream flows at about 160° F. at a rate of about 1168 b./d., containing about 5 t./d. of sand and 1 b./d. of oil. Thickener 16 is an essentially cylindrical vessel provided internally with a coaxial central well 62 into which all of the fluids for treatment are introduced. The floor of thickener 16 is provided with a plurality of radial rake arms 64 rotated by means of a vertical central shaft 66 or by other means, driven by rotating means 68. In the present example the central well is such that the fluid residence time is about one hour devoted to the settling of silt from the oil phase as well as the separation of the oil and water phases.

The annular volume outside well 62 is sized to give a water residence time of about 6 hours during which time substantially all of the silt settles from the aqueous phase. Rake arms 64 are provided with rakes inclined at such an angle so that rotation of the rakes move the settled silt as a thickened sludge radially inward toward silt outlet 70. The thickened silt is removed through line 70 at a rate controlled by valve 72, the silt concentrate containing about 87 b./d. of water and 15.0 t./d. of solids.

The clear water effluent is removed from collector 74 surrounding the upper periphery of thickener 16 by means of line 76 at a rate of 1821 b./d. This material actually constitutes the aqueous silicate solution to which makeup aqueous silicate concentrate is introduced by means of line 78 at a rate of 2.5 gallons per hour controlled by valve 80. Fresh water is introduced by means of line 82 at a rate of about 355 b./d. controlled by valve 84. This may, if desired, flow into the clear aqueous stream in line 76. As previously indicated this is preferably employed, wholly or in part, as wash water for the spent sand and is introduced through line 54 previously described. The total aqueous stream from thickener 16 continues through heat exchanger 30. It is heated to about 180° F. and is introduced into pulping zone 10 through line 26 as previously stated.

The overflow of the wet oil phase from primary separator zone 12 passes through line 86 into central well 62 of thickener 16. This stream flows at a rate of about 1081 b./d. and includes 754 b./d. of water, 327 b./d. of oil, and 12 t./d. of silt and sand. The temperature of the stream is about 175° F.

Also introduced into the central well 62 at a temperature of about 155° F. is a relatively small stream of water from the bottom of settling zone 18. This passes through line 88 into central well 62 and contains 67 b./d. of water, 1 b./d. of oil, and a trace of silt and sand.

In central well 62 broken line 90 indicates the approximate position of the oil emulsion-aqueous phase interface. This is maintained at a distance about two-thirds of the way down in the central well. The aqueous streams flowing through lines 60 and 88 from washer-drier 14 and settling zone 18 respectively are introduced below this level because they contain only slight quantities of oil. The primary separator effluent flowing through line 86 and containing about 30% by volume of oil is introduced above level 90 into the supernatant phase consisting of separated oil and possibly a layer of oil-water emulsion. Preferably the interface denoted by line 90 is detected continuously and the rate of removal of the supernatant wet oil phase from weir box 92 or other removal means is controlled so as to maintain a substantially constant position of the interface. In any event, the residence time for the oil phase is approximately one hour and the wet oil stream is removed from weir box 92 through line 94 at a rate of 409 b./d. controlled by valve 96 or other means. The temperature of this stream is approximately 168° F., and it contains 328 b./d. of oil, 81 b./d. of water, and 2 t./d. of sand.

This wet oil stream is discharged into separator zone 18 by means of distributor 98 disposed in the lower portion of the settling zone. Heating coil 104 is provided within settling zone 18. Preferably the volume of settling zone 18 is sufficient to give the wet oil a residence time of about 12 hours permitting it to separate into dry oil and aqueous phases. The separated aqueous phase is removed from the bottom of settling zone 18 through line 88 and contains a trace of solids, but is otherwise essentially all water. The dry oil is removed from the top of settling zone 18 by means of take-off weir 100. This stream is pumped by means of a pump not shown through line 102 to distillation facilities which may be located at the plant site or at a remote area where it is associated with oil refining facilities for treating the recovered oil. This stream flows at a temperature of about 153° F. and contains 321 b./d. of oil, 2 b./d. of water

and 0.1 t./d. of solids. The effluent dry oil is heated in exchanger means 106 and is distilled in distillation column 108. A stripping gas such as steam is introduced into the bottom of distillation column 108 through line 110 at a rate controlled by valve 112. The overhead vapor flowing through line 118 from still 108 is condensed in condenser 120, part of the condensate is returned through line 122 as reflux, and the remainder is pumped by means of a pump not shown through line 22 into pulping zone 10. The stripped diluent oil-free bitumen is removed through line 114 at a rate of 137 b./d. controlled by valve 116. This product oil has the following properties:

TABLE 1

Product oil characteristics

Viscosity, SUS at 180° F.	50,200
Carbon residue, percent by weight	16.05
Sulfur, percent by weight	4.4
Nitrogen, percent by weight	0.95
Gravity, ° API	4.4

By means of the above described process, bituminous sands are readily treated to effect better than 96% by volume of bitumen contained therein at moderate temperatures and pressures and with only slight consumption of chemicals. The sand discharged from the system contains less than 5 pounds per ton of residual oil.

Referring now more particularly to Figure 2, a vertical cross section of primary separation vessel 12 is shown to illustrate the detail of the internal baffles and oil collection equipment. Pulp inlet 40 opens into the upper end of primary separator vessel 12. The sand outlet 48 opens from the bottom thereof and is provided with valve means 50 which is actuated by valve positioner 120 which in turn is actuated by solids level controller 123. Solids level detecting grid 124 is positioned within the pulp inlet line 40. It detects the presence of a compact bed of solids and controls the rate of solids removal so as to maintain a compact sand bed 128 from 2 to 15 inches thick moving downwardly and outwardly across the surface of the vertical conical primary baffles 126, 128, and 130, and inwardly and downwardly across the upper surface of inverted truncated secondary baffles 132 and 134. In this way the countercurrent flow of the aqueous phase, described immediately below, is filtered through the moving sand bed. This reduces to a minimum the amount of fine solids and silt carried up and out with the fluid oil and aqueous phases.

The downwardly moving sand bed receives moderate physical agitation due to its zig-zag or serpentine path. However, to enhance the agitation and to insure that the downwardly moving bed of sand is disturbed sufficiently to liberate the occluded or mechanically trapped oil particles, fluid inlet 44 and valve 46 are provided. The fluid so introduced is a recirculated stream of the dilute aqueous silicate solution. It is introduced at a rate sufficient to effect a net upward flow of the aqueous phase through primary separator 12, and to agitate the moving bed of sand therein. As shown in Figure 2 the lower peripheries of the primary and secondary baffles are provided with outwardly and inwardly projected louvres respectively. The louvred portion does not exceed about 35% of the slant height of the conical section of the baffle and thus the silicate solution passes through the louvres and then transversely through the downwardly moving solids bed superimposed above the baffle surface. This serves to agitate and sweep out of the disturbed sand bed small globules of oil and carry them upwardly from the sand bed with the aqueous flow into an area below the next superjacent baffle.

As indicated in Figure 2, the primary baffles, as represented by baffle 126 for example, is provided with a flat bottom portion 136 and a central riser portion 138 which opens upwardly to a point just opposite the uppermost louvre. This serves to direct the rising silicate solution and the liberated oil globules centrally into the primary baffle. Due to the flow area enlargement at the outlet of the riser, the liquid velocity is rapidly decreased permitting the oil globules to rise slowly to an oil accumulation just below the apex **138** of the primary baffle. The silicate solution then progresses radially after separation of the oil globules through the louvres **140** and then back through the downwardly moving sand bed to liberate more oil particles.

The secondary baffles, represented by baffle **132** for example, are also provided with a flat portion **142** and a vertical cylindrical riser portion **144**. This cylindrical portion also extends to approximately the top of the louvres and its purpose is the same, namely to permit separation of the oil globules before the silicate solution passes through the next set of louvres and back into the sand bed.

The net effect of the baffle structure shown and described above is the sequential agitation of the downwardly moving sand bed by means of a countercurrent flow of aqueous silicate solution, separation from the silicate solution of any oil globules liberated from the sand, and then passage of the silicate solution again through the next sand bed. These steps are of course repeated in sequence until the silicate solution emerges from the uppermost end of the solids bed.

From the upper portion of each primary baffle lead primary oil outlet lines **150**, **152** and **154**. Correspondingly, secondary oil outlet lines **156** and **158** are provided at the upper ends of secondary baffles **132** and **134**. These lines extend upwardly to a point adjacent oil and water outlet **86** which opens from a point near the top of primary separator vessel **12**. These oil collection lines are each provided with a valve **160** for control purposes and to minimize flow surges. The upper end **162** of each line is disposed at a point above liquid level **164** which is fixed by the position of oil and water outlet **86**. By proper adjustment of the distance of each of the outlet openings **162** above liquid level **164**, the oil collection lines may be made to discharge only oil at a rate equal to the rate that it collects beneath the various baffles, and prevent the bypassing of the upwardly flowing silicate solution therethrough.

First collection line **150** has a liquid pressure imposed upon its entrance which is equal to the hydrostatic head of liquid in separator vessel **12** above the entrance point to liquid level **164**, plus the pressure differential generated by the flow of silicate solution through the solids bed around the uppermost primary baffle **126**. The magnitude of this differential varies with the silicate flow rate and the thickness of the sand bed above each baffle. Preferably a differential of at least 5 inches of water is maintained across each bed. This means then that the liquid contained in first oil outlet line **150** will ordinarily stand in that line to a height above liquid level **164** which is equivalent to this pressure differential generated by the liquid flow through the sand bed, since the hydrostatic head in the separator vessel will be essentially balanced by that in line **150**. This pressure differential may be determined by a differential pressure indicator **174**, or the height may be manually adjusted during operation while an operator watches its operation. In either case, the upper outlet end **162** of first oil collection line **150** is adjusted so as to be at a distance above the liquid level **164** which is less than the hydrostatic head expressed in feet or inches of oil phase equivalent to the pressure drop of silicate flow through the sand bed, and which is greater than the corresponding equivalent hydrostatic head of the aqueous phase. This adjustment is made for each of the oil outlet lines opening from the upper end from each of the primary and secondary baffles, it being noted that the height differences for the successively lower outlet lines are successively greater since the pressure differentials of the liquid flow through the sand beds are additive.

This adjustment and the height differences are more clearly detailed in Figures 3 and 4.

Referring now to Figure 3 the uppermost section of primary separator column **12** is shown together with oil and water outlet line **86** and pulp inlet line **40**. The first four successively lower oil outlet lines are shown as lines **150**, **156**, **152**, and **158**. An adjustable sleeve **170**, enlarged at its upper end to eliminate liquid level fluctuations due to surging in the line, is provided at the upper end of each of these oil collection lines together with adjustment means **172** whereby the height of each oil collection line above liquid level **164** may be varied. In Figure 2 the pressure differential existing across the sand bed from points inside primary baffle **126** and above the superjacent sand bed is indicated by differential pressure indicator **174**. This differential pressure, in pounds per square inch for example, has an equivalent hydrostatic head expressed in inches of the oil phase and also an equivalent head which may be expressed in inches of the aqueous phase. These equivalent heads are of course dependent upon the specific gravity of each of these phases, the inches of head for the aqueous phase being smaller than the inches of head for the oil phase with oil phases of gravity greater than 10° API.

According to the principles of the present invention, the distance at which upper outlet opening **162** of first oil collection line **150** above liquid level **164** is equal to h_1 and this is adjusted by means of a sleeve, not shown but equivalent to **170**, to a value in inches which is greater than the equivalent aqueous head and less than the equivalent hydrocarbon head. Then for a given flow rate of silicate solution through the sand bed generating a given differential pressure, the oil will collect under primary baffle **126** and accumulate in oil outlet line **150** until sufficient oil exists in the collection line to raise the head above outlet opening **162**. At this time only oil will discharge and flow from separator **12** through outlet line **86**. If no oil is collected at this point, the aqueous head equivalent to the existing differential pressure is not sufficient to cause water to overflow from outlet **162**, since the equivalent aqueous head is less than h_1. Therefore only oil, when and if it accumulates in sufficient amounts, will overflow. No aqueous phase can bypass the sand beds through the oil collection lines.

Identical considerations apply to each of the successive 4, 5 or more oil collection lines with successively greater heads h_2, h_3, h_4, etc.

The nature of the fluid overflowing from a given oil collection line is indicated in Figure 4 in the manner of a phase diagram. The equivalent liquid head for the oil and aqueous phases are here related to the total accumulative pressure differential of aqueous silicate flow through the sand beds. The equivalent head of the oil phase is shown by curve **180** and the equivalent head for the aqueous phase is shown by curve **182**. If for a given total effective pressure differential the height differential h_1 is set at a value corresponding to point **184**, then the aqueous head equivalent exceeds the height differential and water and any oil accumulating below the corresponding baffle will overflow at the outlet of each of the oil collection lines. This means that aqueous phase flow can bypass the sand beds through the oil collection lines to the primary separator outlet. If the height differential is set at a value corresponding to point **186**, there will be no fluid overflow at all since the height is greater than the equivalent hydrostatic head expressed in inches of pure oil. However, and this is an essential principle of the present invention, if the height differential is set at a value corresponding to point **188**, i.e., at a value greater than that of the aqueous head but less than that of the oil head, then only oil will overflow since only a hydrostatic head of oil in the collection line will stand sufficiently high to reach to and over the outlet end of the line.

The purpose of the differential pressure determination and the adjustment of the height differential between the liquid level in separator 12 and the upper outlet end of each of the oil collection lines is to prevent aqueous phase bypassing upwardly through the oil collection lines so as to force the aqueous silicate solution to flow in serpentine fashion back and forth through the downwardly moving sand bed while permitting any separated oil to flow through these lines out of contact with the settling sand.

The primary separator equipment of the present invention was applied in the treatment of Sisquoc bituminous sand as described in connection with the preceding Figure 1. The primary separator column was 10 feet high and 22 inches in diameter and was provided with 5 conical baffles substantially as shown in Figure 2. With a flow of bituminous sand of about 172 t./d. downwardly through the separator so as to maintain a dense downwardly moving bed through the separator, an upward silicate flow countercurrent to the sand of about 1300 b./d. was maintained. The height differential of each of the oil collection lines was adjusted visually while the nature of the fluid overflowing was observed. With more or less steady operation an excessively low height resulted in an excessive liquid flow, primarily of aqueous silicate solution, from the outlet of the oil collection line. The height was gradually increased until a steady smooth overflow of oil resulted. The height differentials of all of the lines were similarly adjusted. The sand produced from the bottom of the primary separator contained as low as 2 pounds of residual oil per ton.

A particular embodiment of the present invention has been hereinabove described in considerable detail by way of illustration. It should be understood that various other modifications and adaptations thereof may be made by those skilled in this particular art without departing from the spirit and scope of this invention as set forth in the appended claims.

I claim:

1. In a process for the recovery of hydrocarbon values from naturally-occuring hydrocarbonaceous mineral solids which comprises contacting said solids with an aqueous chemical solution and a hydrocarbon diluent at a moderately elevated temperature for a period sufficient to reduce said solids to a homogeneous fluid pulp, and said pulp is then treated to separate it into a solids phase, a hydrocarbon phase, and an aqueous phase, the method of effecting said separation treatment which comprises: (1) introducing said pulp into the upper end of a confined vertically elongated separation zone, said separation zone comprising within its confines a plurality of vertically spaced hydrocarbon separation zones; (2) allowing the solids components of said pulp to settle into a substantially compact bed at the upper end of said separation zone; (3) allowing said compact bed to descend by gravity through said separation zone to the lower end thereof while directing the path of said descent past said hydrocarbon collection zones and alternately towards the periphery and towards the axis of said separation zone, whereby the solids pass downwardly in a sinuous path through said separation zone in the form of a continuous compact bed having the shape of a vertical sinusoidal envelope; (4) removing essentially hydrocarbon-free solids from the lower end of said separation zone; (5) simultaneously introducing an aqueous liquid into the lower end of said separation zone; (6) passing said aqueous liquid in a continuous stream upwardly through said separation zone and through said hydrocarbon collection zones in a sinuous path which passes transversely across the sinusoidal path of descending compact solids bed; (7) removing an aqueous phase from the upper end of said separation zone; (8) removing a hydrocarbon phase from each of said hydrocarbon collection zones; (9) separately passing the hydrocarbon phase removed from each of said hydrocarbon collection zones in a confined stream out of contact with the compact solids bed and out of contact with said aqueous liquid to a discharge point located a sufficient distance above the liquid level in said separation zone that substantially only hydrocarbon is discharged at said point; and (10) removing the so-discharged hydrocarbon from the upper end of said separation zone.

2. A process as defined by claim 1 wherein said hydrocarbonaceous mineral solids is tar sand.

3. A process as defined by claim 2 wherein the thickness of said downwardly moving compact solids bed is maintained between about 2 and about 15 inches, and the said aqueous liquid is passed upwardly through said separation zone at such a rate that a pressure differential of at least about 5 inches of water is generated each time the said aqueous liquid passes through said descending solids bed.

4. A process as defined by claim 2 wherein the said chemical solution and said aqueous liquid consist essentially of an aqueous solution of sodium silicate in which the Na_2O/SiO_2 ratio is at least above about 0.4.

5. The process for recovering hydrocarbon values from bituminous sands which comprises (1) agitating said sand at a temperature between about 160° F. and about 250° F. with between about 0.1 and 2.5 barrels per ton of a relatively light hydrocarbon oil and with between about 0.75 and about 5.0 barrels per ton of an aqueous sodium silicate solution for a period of time sufficient to form a substantially homogeneous fluid pulp, said sodium silicate solution containing between about 0.5 and about 20 pounds per barrel of an aqueous sodium silicate concentrate which contains about 34 percent by weight of a sodium silicate having a Na_2O/SiO_2 ratio of at least about 0.5; (2) introducing said pulp into the upper end of a confined vertically elongated separation zone; (3) allowing the solid components of said pulp to settle into a substantially compact bed at the upper end of said separation zone; (4) allowing said compact bed to descend by gravity through said separation zone while directing the path of said descent alternately to the periphery and to the axis of said separation zone a plurality of times, whereby the solids pass downwardly in a sinuous path through said separation zone in the form of a continuous compact bed having the shape of a multi-noded vertical sinusoidal envelope having walls between about 2 and about 15 inches thick; (5) removing essentially hydrocarbon-free solids from the lower end of said separation zone; (6) simultaneously introducing aqueous sodium silicate into the lower end of said separation zone; (7) passing said aqueous sodium silicate in a continuous stream upwardly through said separation zone in a sinuous path which passes transversely through the walls of the aforesaid envelope of descending solids; (8) removing an aqueous phase from the upper end of said separation zone; (9) collecting a hydrocarbon phase at each of a plurality of points within said separation zone, each of said collection points being located within said envelope of descending solids at top of each node of said envelope; (10) separately passing the hydrocarbon phase collected at each of said collection points in the form of a confined stream out of contact with the descending solids and out of contact with said aqueous sodium silicate to a discharge point located a sufficient distance above the liquid level in said separation zone that substantially only hydrocarbon is discharged at said point; and (11) removing the so-discharged hydrocarbon from the upper end of said separation zone.

6. An apparatus for separating a fluid pulp comprising mineral solids, liquid hydrocarbons and water which comprises a vertically elongated vessel closed at its upper

end and lower end and having a liquid outlet opening through its wall adjacent to its upper end and a solids outlet opening adjacent its lower end; a pulp inlet conduit extending into said vessel adjacent its upper end and terminating within said vessel at a point below said outlet opening; a plurality of primary baffles positioned in spaced relationship within said vessels, each of said primary baffles taking the form of a hollow cone whose axis coincides with that of said vessel, whose apex is directed upwardly, whose lower periphery has a diameter less than the internal diameter of said vessel, and having louvres opening through its wall adjacent the lower edge thereof; a plurality of secondary baffles positioned in spaced relationship within said vessel and alternation with said primary baffles, each of said secondary baffles taking the form of a hollow inverted truncated cone whose axis coincides with that of said vessel, whose upper peripheral edge is in register with the wall of said vessel, and having louvres opening through its wall adjacent the lower edge thereof; a plurality of first conduit means each of which extends through the wall of one of said primary baffles adjacent the apex thereof and terminates at a point above said outlet opening; a plurality of second conduit means each of which extends from a point adjacent and below the upper edge of one of said secondary baffles and terminates at a point above said outlet opening; and a liquid inlet conduit extending through the wall of said vessel and terminating within said vessel at a point below the lowermost of said baffles.

7. An apparatus as defined by claim 6 wherein the uppermost of said baffles is a primary baffle having its apex disposed immediately below the opening of said pulp inlet conduit, and the lowermost of said baffles is a primary baffle having its apex disposed immediately above the opening of said liquid inlet conduit.

8. An apparatus as defined by claim 6 wherein each of said primary baffles is provided with an annular plate having its outer edge in register with the lower edge of the baffle and an open-ended cylindrical element extending from the inner edge of said plate upwardly to a level adjacent that of the uppermost of the louvres of said primary baffle and each of said secondary baffles is provided with an annular plate having its inner edge in register with the lower edge of the baffle and an open-ended cylindrical element extending from the outer edge of said plate upwardly to a level adjacent that of the uppermost of the louvres of said secondary baffle.

9. An apparatus as defined by claim 6 wherein means are provided for adjusting the levels at which said first and second conduit means terminate above said outlet opening.

10. An apparatus as defined by claim 9 wherein said level adjusting means comprises a vertically movable sleeve disposed in liquid receiving relationship at the upper end of each of said first and second conduit means, a substantial part of said sleeve having a diameter substantially larger than that of the conduit means with which it is associated.

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