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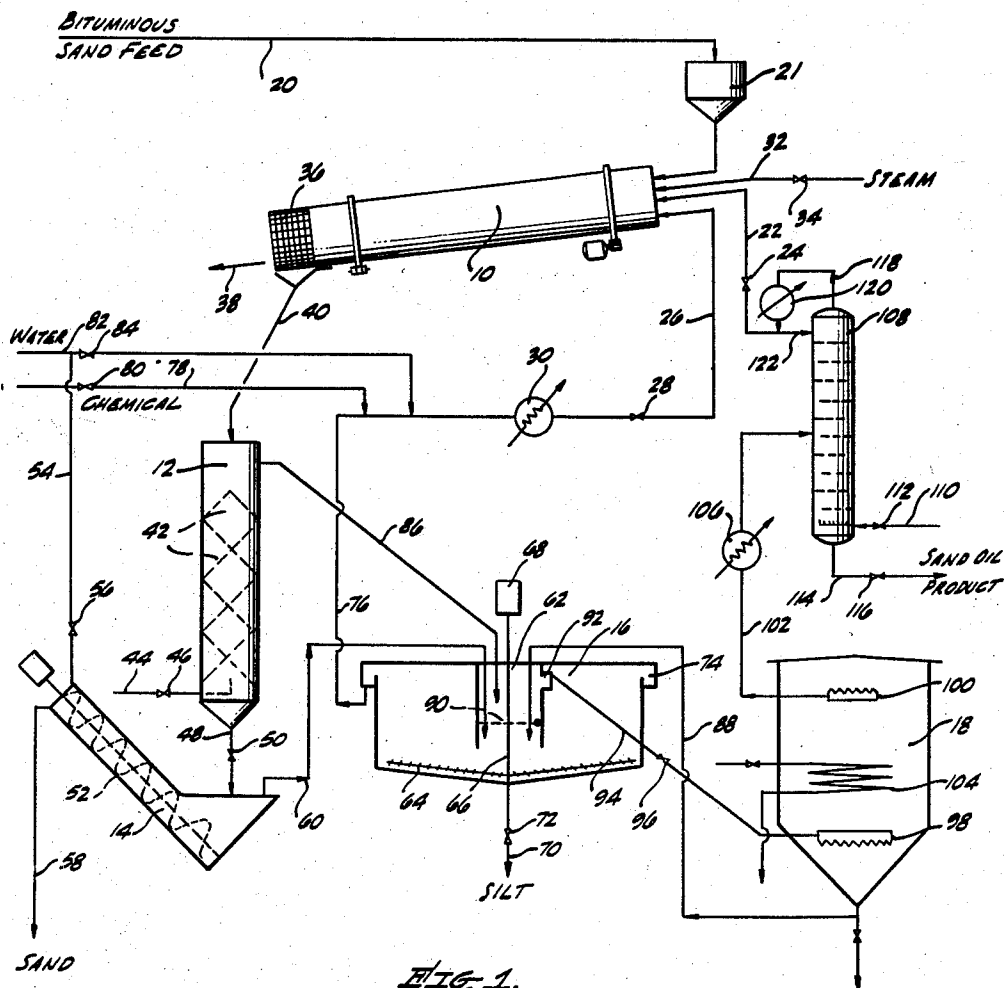
R. P. VAELL ET AL

**2,924,566**

## TREATMENT OF BITUMINOUS SANDS

Filed July 26, 1957

2 Sheets-Sheet 1



*INVENTOR.*

RAOUL P. VAELL  
PAUL W. FISCHER

BY  
Richard C. ~~Sutcliffe~~  
ATTORNEY.

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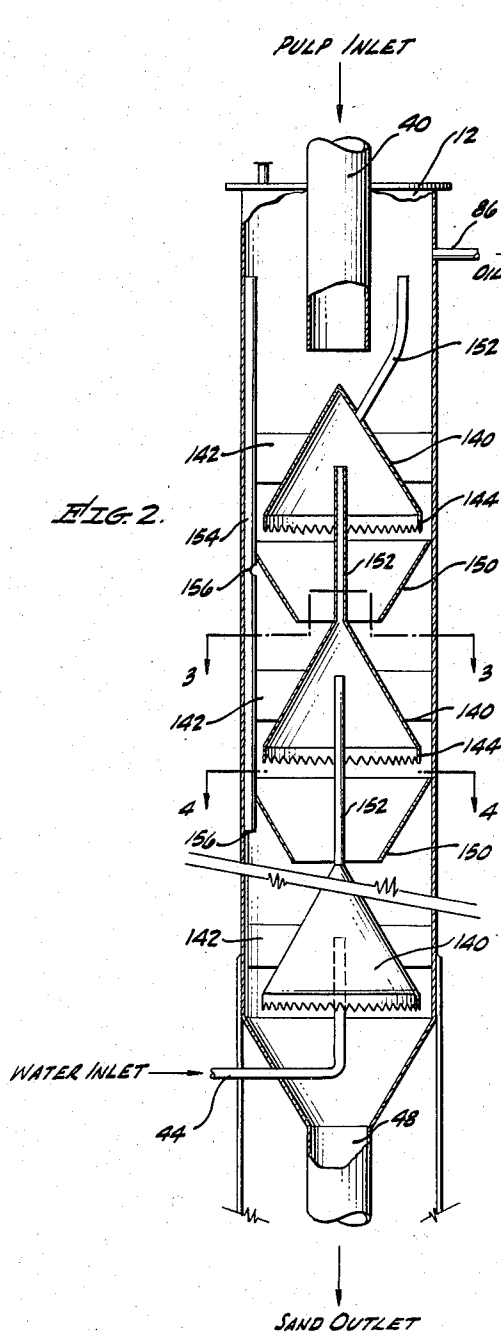
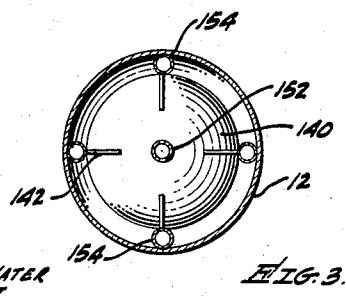
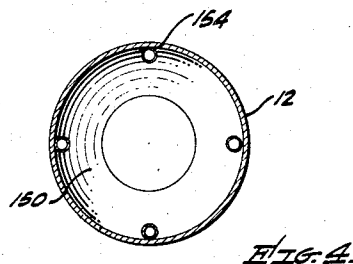
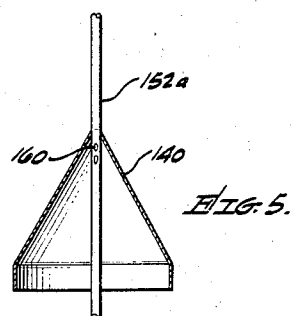
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TREATMENT OF BITUMINOUS SANDS

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2 Sheets-Sheet 2



INVENTORS.  
RAOUL P. VAELL,  
PAUL W. FISCHER,

BY *James J. James*  
ATTORNEY.

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2,924,566

## TREATMENT OF BITUMINOUS SANDS

Raoul P. Vaell, Hollywood, and Paul W. Fischer, Whittier, Calif., assignors to Union Oil Company of California, Los Angeles, Calif., a corporation of California

Application July 26, 1957, Serial No. 674,508

13 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 and which may utilize particularly efficient storage pretreatment together with sand 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 a silicious matrix, 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 out-croppings. 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 revealed 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 lay in 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 and is based upon 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 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 hydrocarbon coating.

The present invention is directed to a low temperature

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process using a warm aqueous solution of a special alkali metal silicate, with or without other reagents, and a relatively light 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 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 phase, 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, in its preferred modification, the mining of surface or near-surface deposits of tar sand and the like by the usual procedures to produce a raw feed material consisting of chunks or pieces of tar sand of the order of about 15 inches in average dimension. Smaller sizes digest faster, but the sizes may be larger in some cases. This may be done by open pit mining in which overburden is stripped away and the tar sand is mined by means of bull dozers, clam shell shovels, and similar equipment. Drilling and blasting may also assist in the breaking up of the tar sand into the aforementioned sized particles. The mined material is fed 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 with internal baffles and conveyor flights so as to control the duration 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 during the dropout of the sand grains from the fluid phases in order to liberate residual oil droplets which are trapped in the downwardly progressing sand. 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 hydrocarbon 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 are sent, a substantially

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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. The aqueous phase containing the silt is recirculated from the wash tank 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 prompt treatment of the mined bituminous material and 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 in which the sand is agitated and separated from all oil,

Figures 3 and 4 show transverse cross sections of the apparatus of Figure 2, and

Figure 5 shows an elevation view in partial cross section of a modified conical baffle which can be used in the apparatus of Figure 2.

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 of from 2 to 10° API, a typical bituminous sand contains about 30 gallons per ton of 4° API 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 of the foregoing ingredients 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° and is preferably maintained between limits of about 0.1 and about 2.5 b./t. (barrels per ton) of raw bituminous sand feed. The

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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. 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<sub>2</sub>O to SiO<sub>2</sub> 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 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 33 separately. 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. If desired, additional agitation may be 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. The rate of flow of sand is controlled by controlling the flow of pulp into the separation zone so that no substantial downwardly moving bed of sand forms therein.

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 b./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 1169 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 6-8 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. This is sent to a tailings pond from which any recoverable aqueous liquid is returned to the process, as by pumping back into line 60 and the thickener.

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 partly, 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 also into central well 62 of thickener 16. Emulsion breaking chemical reagents may be added to this overflow stream if needed. 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, together with makeup diluent oil added by means of a line not shown. 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 I

## 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 recovery of better than 96% by volume of the 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, primary separator 12 is provided with pulp inlet line 40, oil and water outlet line 86 opening from the upper end of column 12, and a lower sand outlet opening 48 provided with a valve 50 not shown but indicated in Figure 1. Superimposed above one another and supported from the inside surface of vessel 12 is a series of conical primary baffles 140 spaced apart from one another coaxially within vessel 12. They are supported by means of one or more gusset plates 142 hereinafter more fully illustrated and described. Preferably the lower periphery of each primary baffle is provided with a notched or sawtoothed edge 144. Disposed between the various primary conical baffles 140 are inverted truncated secondary baffles 150. The upper major base of these conical baffles is connected at the inner wall of column 12 and arranged coaxially with the vessel and the primary baffle axis.

The conical baffles are each geometrically a right circular cone with an apex angle of between about 40° and about 100°. The downward slope from a horizontal plane of the surface of the conical baffles therefore is between about 40° and 70°. Too steep an incline unnecessarily increases the height of the apparatus for a given number of baffles. Too low a slope causes the solids to tend to settle on the baffle surfaces and inhibits their downward flow. Accordingly apex angles and slopes within the limits named must be employed. The internals of separator vessel 12 thus consist of a series of alternate conical primary baffles and inverted truncated conical secondary baffles coaxially superimposed one above the other. The solid material in the pulp, which discharges through line 40 into the top of vessel 12, moves in a zig-zag or serpentine fashion alternately outwardly to form a thinner solids bed near the lower edge thereof and downwardly across the surface of

primary baffles and then downwardly and inwardly across the surface of the secondary baffles reforming the thicker solids bed near the lower edge thereof.

In order to enhance the gentle agitation of the sand grains as they settle downwardly across the baffle surface, fluid inlet 44 is provided for the introduction of wash water, which in this process is preferably a recirculating stream of aqueous silicate solution. This material is discharged below the lowermost primary baffle and progresses upwardly in the reverse serpentine flow. At the peripheries therefore of each of the baffles, where the solids fall from the lower edge of one baffle to the upper surface of the next, turbulence is increased whereby sand grains are separated from one another and the liberated oil globules tend to rise in and with the rising aqueous phase. However, below each of the primary and secondary baffles is a quiet collecting space through which there is little if any net flow of aqueous solution and in which the liberated oil globules are permitted to rise and separate to form a separate oil phase. Each of the primary baffles 140 is provided with an upwardly extending open-ended primary oil outlet conduit 152 which opens upwardly into the region below the next superjacent primary baffle. The only exception is in the case of the uppermost primary baffle in which case the conduit extends upwardly offset from the pulp inlet 40 into the supernatant liquid surrounding the pulp inlet at which point the oil discharges into a region adjacent outlet line 86.

Droplets of oil liberated in the same manner also collect in the annular spaces surrounding and below the lower surface of the secondary baffles 150. This oil is best collected by means of at least one secondary oil outlet or manifold line 154. This line extends up and along the inside wall of separator 12 and terminates in the same region as does the uppermost primary oil outlet line 152. The secondary manifold is provided with openings 156 immediately below each of the superjacent secondary baffles 150. Oil collecting in these places rises through manifold 140 and is discharged into the top of separator 12. In this way oil droplets liberated by the gentle gravity agitation and the countercurrent aqueous solution flow remain separated from the downwardly flowing sand current and the upwardly flowing aqueous phase. Rewetting of the sand grains with the oil is prevented.

Referring now more particularly to Figure 3, a cross section view of separator vessel 12 is shown including the upper surface of a primary baffle 140 and showing the upwardly extending primary oil outlet 152. Disposed at 90° intervals around the periphery of the primary baffle 140 is a plurality of four secondary oil outlet lines 154. Gusset plates 142 are also shown.

Figure 4 is an analogous cross sectional view of one of the secondary baffles 150. From Figures 3 and 4 the construction of the internals and their means of support within primary separator vessel 12 is clear.

Referring finally to Figure 5, a somewhat modified form of primary baffle 140 is shown in which the coaxial primary oil outlet line is somewhat modified into a continuous centrally disposed pipe 152a which opens through a series of apertures 160 at a point immediately below each apex of the interconnected primary baffles. Line 152a thus becomes a means for supporting the primary baffles within the column.

The apparatus of the present invention was applied in the primary separation of the pulped tar sand mined near Sisquoc in Southern California under conditions essentially the same as those given in the foregoing description of Figure 1. The primary separator was provided with a plurality of 5 superimposed primary and secondary baffles substantially as shown in Figure 2. The column was 22 inches in diameter and 10 feet high and the primary and secondary outlet lines were 1 inch nominal iron pipe size. Pulp was introduced at a rate of 144 b./d.

at a temperature of about 180° F. and the aqueous agitation or wash fluid consisted of 180° F. aqueous silicate introduced through line 44 at a rate which was varied between about 160 and about 500 b./d. This is sufficient to give an upward fluid velocity around the primary baffle peripheries of between about 0.5 and about 1.6 feet per minute. Whereas the spent sand is contaminated with about 15 to 20 pounds of residual oil per ton as the pulp is discharged merely into a water-filled tank, the spent sand discharged from the primary separator of this invention as shown in Figure 2 contained less than about 3 pounds per ton of sand. This constitutes a 96% recovery of the bitumen contained in the raw bituminous sand feed.

In the process of this invention the solids rates in the primary separation zone are to be maintained between about 10 and about 250 tons per day per square foot of minimum open area at the baffle periphery. Preferably values of between about 20 and 100 tons per day per square foot are maintained. These are controlled by controlling the pulping rate.

The countercurrent flow of aqueous liquid is preferably at a lineal velocity through the smallest open area, i.e. at the baffle peripheries, of between about 0.1 and 5 feet per minute. This corresponds to a liquid flow rate of between about 25 and about 1200 barrels per square foot minimum open area per day. Under these conditions the solids are substantially completely freed of mechanically trapped oil.

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.

We claim:

1. In a process for the recovery of hydrocarbon values from petroliferous solids wherein (1) said solids are treated with an aqueous sodium silicate solution and a relatively light hydrocarbon diluent at a moderately elevated temperature for a period of time sufficient to reduce the solids to a substantially homogeneous pulp, (2) said pulp is treated to separate it into a solid phase and a heterogeneous liquid phase essentially comprising liquid hydrocarbons and aqueous sodium silicate solution, and (3) said heterogeneous liquid phase is then treated to separate said aqueous solution and said liquid hydrocarbon, the method of effecting said step (2) which comprises introducing said pulp into a vertical elongated separation zone at a point adjacent the top thereof; allowing the solid components of the pulp to flow downwardly by gravity through said separation zone while directing said downward flow alternately towards the periphery and towards the central axis of said separation zone, introducing an aqueous liquid into said separation zone at a point adjacent the bottom thereof; flowing said liquid upwardly through said separation zone while directing said upward flow alternately towards the periphery and towards the axis of said separation zone essentially parallel and countercurrent to said downward flow of solids, removing substantially hydrocarbon-free solids from said separation zone at a point adjacent the bottom thereof, collecting liquid hydrocarbon at a plurality of points spaced vertically along the lengths of said separation zone, flowing the liquid hydrocarbon so collected upwardly into the top of said separation zone independent of the upflow of said aqueous liquid, and removing a mixed liquid phase comprising hydrocarbons and aqueous liquid from the top of said separation zone at a point above that at which said pulp is introduced into said separation zone, while maintaining said separation zone substantially filled with liquid during the aforesaid steps.

2. A process according to claim 1 wherein said petrolif-

erous solid is bituminous sand and, in step (1), the temperature is maintained between about 160° and about 250° F., between about 0.1 and 2.5 barrels of hydrocarbon diluent and between about 0.75 and about 5.0 barrels of aqueous sodium silicate solution are employed per ton of said sand, and said aqueous sodium silicate solution contains between about 0.5 and about 20 pounds per barrel of a 34 percent by weight aqueous sodium silicate concentrate in which the  $\text{Na}_2\text{O}$ -to- $\text{SiO}_2$  ratio is greater than 0.4.

3. A process according to claim 1 in combination with the step of contacting the substantially hydrocarbon-free solids removed from said separation zone with a counter-current flow of makeup water to said process thereby removing residual sodium silicate solution from said solids, and recirculating the wash water so produced in the process.

4. A process according to claim 1 in combination with the steps of passing said mixed liquid phase removed from said separation zone from said primary separation zone to a thickening zone, separating a wet oil phase from said aqueous phase therein, permitting the aqueous phase to stand so as to settle substantially all of the silt-like solids therefrom, removing a clear aqueous stream and a thickened silt slurry separately from said thickening zone, drying said wet oil phase, and separating said diluent oil therefrom for recirculation from the recovered petro-  
liferous material.

5. A process according to claim 2 in combination with the step of controlling the pulp flow rate into said separation zone so as to limit flow of said sand to between about 20 and about 100 tons per day per square foot based on the minimum transverse area open to flow in said separation zone, and controlling the flow of said aqueous liquid at a lineal velocity of between about 0.5 and about 1.6 feet per second through said minimum area.

6. A process according to claim 1 in combination with the step of controlling the rate of pulp introduction into said separation zone so that no substantial downwardly moving bed of sand forms therein.

7. A process according to claim 1 in combination with the step of maintaining a mass rate of solids flow downwardly in said separation zone between about 10 and about 250 tons per day per square foot based on the minimum transverse area open to solids flow.

8. A process according to claim 1 in combination with the step of controlling the upward flow of said aqueous liquid so as to maintain a lineal velocity of between about 0.1 and about 5 feet per minute based on the minimum transverse area open to liquid flow.

9. An apparatus for separating solids from a substantially homogeneous pulp comprising mineral solids, liquid hydrocarbons and aqueous sodium silicate solutions, which apparatus comprises an elongated vertical vessel having a liquid outlet adjacent the top thereof and a solids outlet adjacent the bottom thereof; a pulp inlet

conduit terminating within said vessel at a point below said liquid outlet; a plurality of conical primary baffles disposed within said vessel in coaxially aligned spaced relationship, the apex of each of said conical baffles being directed upwardly and the lower periphery of said baffles being of such diameter as to allow solids to pass between said periphery and the vertical wall of said vessel; a plurality of truncated inverted conical secondary baffles disposed within said vessel in coaxially aligned spaced relationship, each of said secondary baffles being positioned between adjacent primary baffles and having its larger upper periphery in register with the walls of said vessel; at least one liquid oil collection manifold extending vertically along the wall of said vessel upwardly to a point between said pulp inlet conduit and said liquid outlet, said manifold opening into the space below the upper periphery of each of said secondary baffles; an oil collection conduit communicating between the upper part of the uppermost of said primary baffles and a point between said pulp inlet conduit and said liquid outlet; conduit means placing the spaces below the primary baffles in flow communication with each other; and a liquid inlet conduit terminating within said vessel at a point below the lowermost of said primary baffles.

10. An apparatus according to claim 9 wherein said conical baffles have apex included angles of between about 40° and about 100°.

11. An apparatus according to claim 9 in combination with a cylindrical section having a lower sawtooth edge extending downwardly from the periphery of each of said primary baffles.

12. An apparatus according to claim 9 wherein the said conduit means comprises an open-ended oil collection conduit opening upwardly from the apex of all but the uppermost primary baffle and terminating at a point below the next superjacent primary baffle.

13. An apparatus according to claim 9 wherein the said conduit means comprise an open-ended oil collection conduit extending coaxially through the apices of all of the primary baffles below the highest one, said conduit being provided with an opening in its wall at a point just below each of said apices.

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