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TREATMENT OF BITUMINOUS SANDS

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2 Sheets-Sheet 1

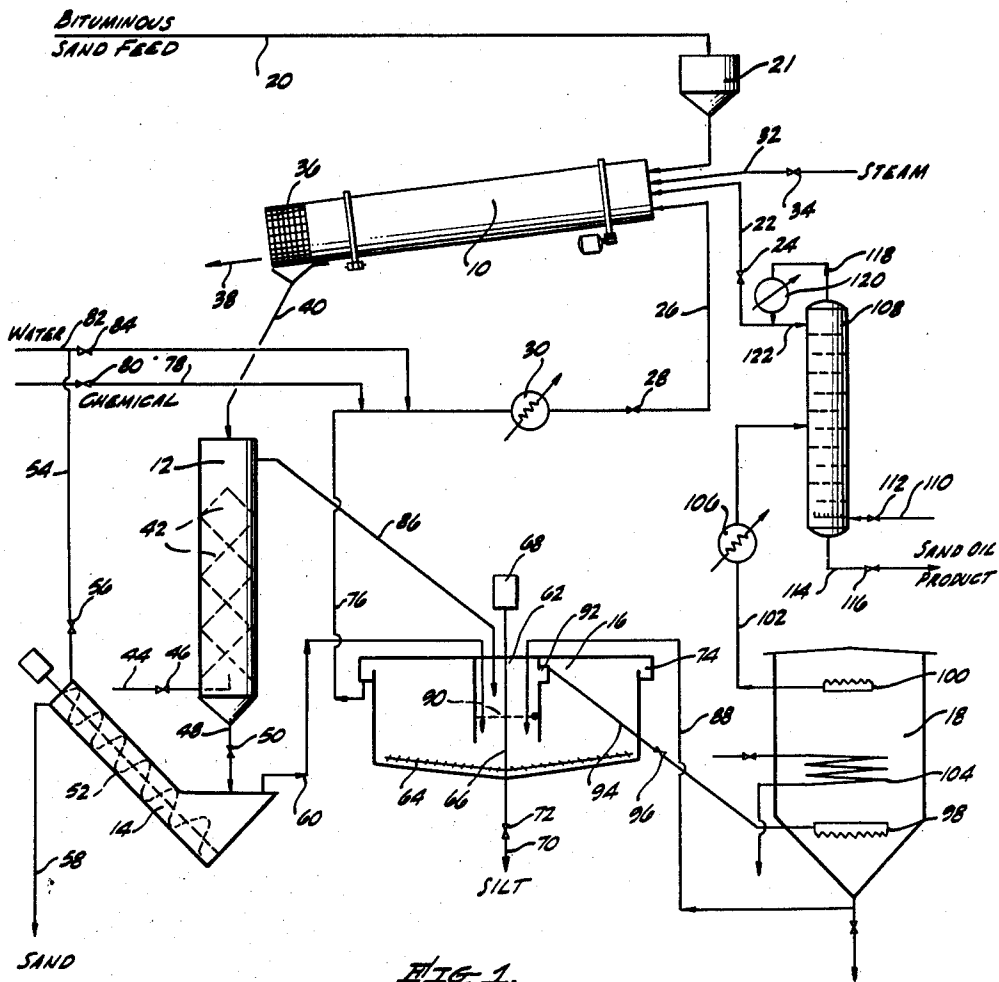


FIG. 1.

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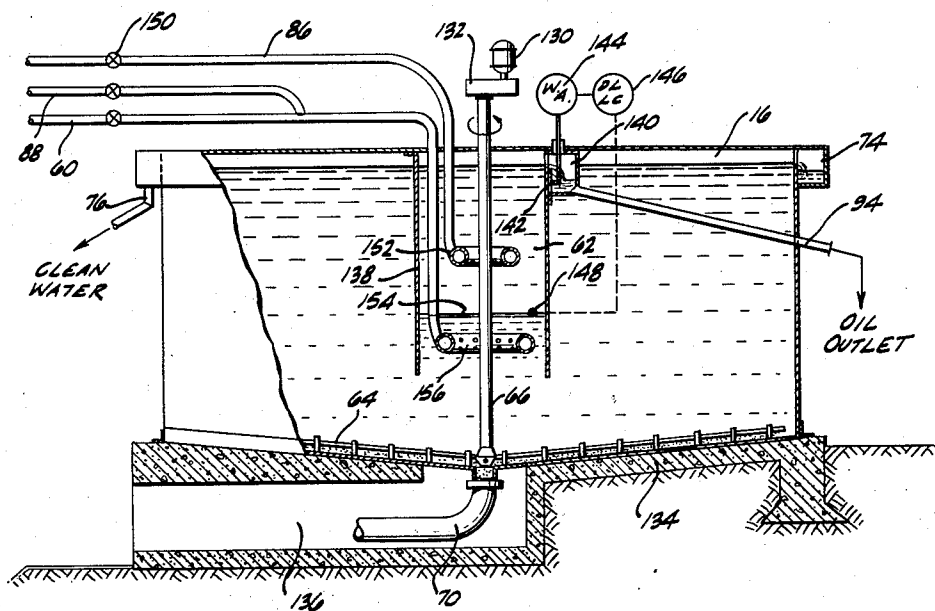


FIG. 2.

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2,921,010

TREATMENT OF BITUMINOUS SANDS

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7 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 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 material, 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 the 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 of 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 tar sand recovery processes lies in the extensive requirement of chemical reagents 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.

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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 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 provide a particular step for treating the oil and water stream which flows from the pulper after removal of most of the solid material whereby a substantially complete separation of fine solids and silt is effected simultaneously from both the aqueous and the hydrocarbon phase.

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 15 inches in average dimension. Smaller sizes digest more rapidly, 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 bulldozers, 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 transferred to 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 residence time of the material in the mixer. This treatment reduces the tar sand chunks to a heavy slurry 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 which includes 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 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 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 substantially dry oil phase is discharged from the thickener into a settling zone, such as a wash tank, if desired, to complete removal of traces of water. This is an optional step in this invention. Otherwise, the oil product is essentially water and silt free, 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. From the bottom of 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 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, and

Figure 2 is an elevation view in cross section showing the details of the thickening zone into which the oil and aqueous effluent from the primary separator zone is discharged for complete separation of the aqueous, hydrocarbon, and silt phases.

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, and thickener 16. An optional oil product storage-settler 18 may be used if desired. 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 an aqueous alkali metal silicate solution, with or without other reagents, 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 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 Na₂O to SiO₂ 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 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. If desired additional agitation may be provided by introducing fluid such as recycle silicate solution 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 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 make up 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.

The overflow of oil and water from primary separator zone 12 passes through line 86 also 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.

Thickener 16 is an essential cylindrical vessel provided internally with a coaxial central well 62 into which all of the fluids for treatment are introduced. In thickener 16 a complete separation of silt-like solids from the

oil and aqueous phases, and the oil and aqueous phases from each other are effected. 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 12 hours 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 6.0 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 17.0 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 make up 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 where it is heated to about 180° F. and is introduced into pulping zone 10 through line 26 as previously stated.

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 the optional 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 and the rate of removal of the supernatant water and silt-free 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 12 hours, during which time the silt and water settle out. The oil stream is removed from weir box 92 through line 94 at a rate controlled by valve 96 or other means. The temperature of this stream is approximately 168° F., it flows at a rate of 328 b./d. of oil, and contains only traces of sand and water.

This essentially dry silt-free oil stream may be discharged into optional storage zone 18 by means of distributor 98 disposed in the lower portion of the vessel. Any separated traces of the aqueous phase are removed from the bottom of settling zone 18 through line 88 and may contain a trace of solids. Heating coil 104 is provided within settling zone 18. The dry oil is removed from the top of settling zone 18 by means of take-off weir 100.

This oil stream is pumped by means of a pump not shown from weir 100 when tank 18 is employed, or otherwise directly from line 94 into and through line 102 to distillation facilities which may be located at the plant site or at a remote area where it is associated with refining facilities for treating the recovered oil. This stream flows at a temperature of about 153° F. and contains 328 b./d. of oil and only traces of water and 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 I

Product oil characteristics

15	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 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, an enlarged detailed view of thickener 16 shown in Figure 1 is here shown in partial cross section. It comprises an inner stratification zone contained in central well 62 and a clarification zone 63 surrounding it. Overflow weir box 74 extends around at least part of the upper periphery of the clarification zone. Drive means 68 of Figure 1 is shown consisting of an electric motor 130, and a reduction gear 132 from which central shaft 66 runs downwardly to connect to radial rake arms 64.

Concrete foundation 134 is provided with a tunnel 136 through which outlet line 70 passes from a central connection at the bottom of vessel 16. This tunnel permits the bottom of the thickener to be opened if necessary and the accumulated silt discharged with the aid of jets of water.

Central well 62 is positioned coaxially within vessel 16 and is contained within cylindrical section 138 which extends downwardly from the roof of vessel 16 a distance equal to between about 50% and 90% of the vessel height. A weir box 140 provided with a variable weir 142 is provided at the upper edge of central well 62. A weir activator 144 which in turn is actuated by differential liquid level controller 146 is provided. The controller is responsive to water-oil interface detector 148. Oil outlet line 94 opens downwardly from the bottom of weir box 140 for the discharge of the dry and silt-free oil collected in central well 62.

Line 86 leading from the top of primary separator 12 and provided with valve 150, opens downwardly into the top of thickener 16 within central well 62 and terminates in a distributor 152 if desired, but in any event located at a point above interface 154. This is the effluent from the primary separator and contains a substantial quantity of oil. By introducing it at a point above interface 154, a residence time of about 12 hours is provided wherein the water and silt drop out substantially completely and pass downwardly through interface 154 and with the aqueous phase into the surrounding body of water in the separator.

Distributor 156 is provided within central well 62 at a point below interface 154. This distributes the essentially aqueous stream containing silt and which also may contain some oil. This stream is the sand washer-drier effluent which is introduced through line 60. Optionally an aqueous stream from the storage zone 18 discharges through line 88. If desired, these may be introduced into central well 62 by means of separate lines if desired and each may be provided with its own distributor. In any

event, any aqueous silt-containing streams which also may contain minor amounts of oil are introduced below interface 154, while the essentially oily streams containing substantial quantities of oil together with silt and solids are introduced above interface 154.

The lower interface 154 is continuously detected by means of element 148 and the maintenance of this interfacial position prevents the flow of oil from central well 62 outwardly around the periphery of cylindrical section 138 into the surrounding aqueous body of fluid in separator 16. With sufficient residence time in central well 62, of 12 hours in this case, the oil overflow through line 94 is dry silt-free oil. Similarly a sufficient residence time of about 20 hours produces silt-free aqueous silicate solution from the upper periphery of the annular shaped surrounding volume of water in separator 16.

The purpose of central well 62 is essentially three-fold: first, to separate substantially all of the silt and solids from the oil phase, second, to separate the oil phase completely from the aqueous phase, and third, to prevent excessive contact of the oil with air in order to avoid degradation of this highly reactive oil. The surrounding annular space in thickener 16 is purely to provide settling time for the aqueous silicate phase in order to remove silt and other solids therefrom. The aqueous phase discharges centrally at the bottom of thickener 16 and flows slowly upwardly to the upper peripheral water drawoff 74. Ample time is thus provided for the gravitational settling of the silt and other solids. As stated previously the clear aqueous phase overflow discharges through line 76 while the settled silt is thickened and forms a heavy slurry which is moved radially inward by rakes 64 to the central silt outlet line 70.

In the experimental verification of the process of this invention, 200 t./d. of Sisquoc bituminous sand were treated according to the process described in Figure 1. The thickener was a vessel 20 feet in height and 30 feet in diameter. The central well was 15 feet in height and 8 feet in diameter and interface 154 was detected and maintained at a point 12 feet from the top of the vessel. The residence time for the oil phase was approximately 12 hours at a liquid feed rate into the thickener equal to those given in the description of Figure 1. The total thickener feed was 415 t./d. or 2408 b./d. including 329 b./d. of oil, 1989 b./d. of water, and 17.0 t./d. of silt and solids. The clear water effluent from the thickener amounted to 1902 b./d. with a negligible quantity of oil and silt. The silt concentrate was composed of 87 b./d. of water and 17 t./d. of solids. The oil overflow from the central well contained 328 b./d. of oil, with only traces of water or silt and solids.

The operating conditions must be varied somewhat to handle mixtures containing silt of various sizes, the finer sizes taking longer to settle. Accordingly in the central well where the oil is freed of water and silt, residence times of from 5.0 to 25 hours and preferably from about 10 to 20 hours for the oil are contemplated. The well should be sized to maintain upward oil velocities on the order of 0.1 to 5.0 feet per hour, and preferably from about 0.25 to 2.0 feet per hour. In the surrounding annular water settling zone, water residence times of from 1.0 to 20 hours and preferably between about 2 to about 10 hours are contemplated. The annular zone should be sized so as to give upward water velocities of between 0.1 and 10.0 feet per hour, and preferably between about 0.5 and about 4.0 feet per hour.

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-occurring hydrocarbonaceous mineral solids wherein said solids are agitated in a pulping zone with aqueous sodium silicate and a liquid hydrocarbon diluent at a moderately elevated temperature for a period of time sufficient to form a substantially homogeneous fluid pulp, and said pulp is thereafter treated to separate therefrom a hydrocarbon phase, an aqueous phase, and essentially oil-free solids, the method of effecting said separation which comprises: (1) introducing said pulp into the upper end of a vertically elongated primary separation zone; (2) allowing the solids components of said pulp to descend by gravity through said separation zone to the lower end thereof; (3) passing a stream of aqueous sodium silicate upwardly through said separation zone in intimate countercurrent contact with the descending solids therein; (4) withdrawing a liquid phase comprising hydrocarbons and aqueous sodium silicate from the upper end of said separation zone; (5) removing de-oiled solids from the lower end of said separation zone; (6) washing said de-oiled solids with water to obtain substantially oil-free solids and a wash liquor comprising water, silt, and hydrocarbons; (7) introducing said wash liquor into the lower part of a stratification zone which opens downwardly into a surrounding clarification zone; (8) introducing the liquid phase obtained in step (4) into the upper part of said stratification zone; (9) maintaining the liquid within said stratification zone in a quiescent state, whereby it separates into an upper hydrocarbon phase and a lower aqueous phase; (10) withdrawing said upper hydrocarbon phase from the upper end of said stratification zone and withdrawing said lower aqueous phase from the upper end of said clarification zone at such rates, relative to each other, that the interface between said phases is maintained at a level between the levels at which the said liquid phase and said wash liquor are introduced into said clarification zone; and (11) withdrawing a thickened slurry of silt from the bottom of said clarification zone.

2. A process as defined by claim 1 wherein the said sodium silicate solution contains between about 0.5 and about 20 pounds per barrel of a 34 percent by weight aqueous concentrate of sodium silicate having a $\text{Na}_2\text{O}/\text{SiO}_2$ ratio greater than about 0.4.

3. A process as defined by claim 2 wherein between about 0.75 and about 5.0 barrels of said aqueous sodium silicate and between about 0.1 and about 25 barrels of said hydrocarbon diluent are employed per ton of said hydrocarbonaceous solids.

4. A process according to claim 1 wherein in step (10), the rate at which said upper hydrocarbon phase is withdrawn from said stratification zone is so controlled relative to the volume of said zone and the rate at which hydrocarbons are introduced therinto that the upward lineal velocity of hydrocarbons within said zone is between about 0.25 and about 2.0 feet per hour, and the residence time of hydrocarbons within said zone is between about 10 and about 20 hours.

5. A process according to claim 1 wherein in step (10) the rate at which said lower aqueous phase is withdrawn from said clarification zone is so controlled relative to the volume of said zone and the rate at which aqueous liquid is introduced therinto that the upward lineal velocity of aqueous liquid within said zone is between about 0.5 and about 4.0 feet per hour, and the residence time of aqueous liquid within said zone is between about 2 and about 10 hours.

6. A process as defined by claim 1 in combination with the steps of introducing the hydrocarbon phase which is withdrawn from said stratification zone into a final settling zone; withdrawing settled water from the bottom of said settling zone; and introducing said water into said stratification zone at a level below that of said interface.

7. A process as defined by claim 1 wherein the aqueous phase withdrawn from said clarification zone is intro-

duced into said pulping zone as said aqueous sodium silicate.

References Cited in the file of this patent

UNITED STATES PATENTS

Re. 21,725	Harrington	Feb. 25, 1941
1,497,607	Streppel	June 10, 1924
2,303,717	Arveson	Dec. 1, 1942

5

2,364,453
2,409,596
2,453,060
2,531,365
2,825,677

563,883

Layng et al.	Dec. 5, 1944
Simpson et al.	Oct. 15, 1946
Bauer et al.	Nov. 2, 1948
Simpson et al.	Nov. 21, 1950
Coulson	Mar. 3, 1958

FOREIGN PATENTS

France	Oct. 5, 1923
--------	--------------