METHOD OF REMOVING SOLIDS FROM BITUMEN FROTH

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Filed: Dec. 16, 2008

Related U.S. Application Data
Provisional application No. 61/066,183, filed on Feb. 19, 2008, provisional application No. 61/065,371, filed on Feb. 11, 2008.

Publication Classification
Int. Cl. C10G 1/04 (2006.01)
U.S. Cl. 208/391

ABSTRACT
A paraffinic solvent, gravity-based process is disclosed for removing solids content from bitumen froth streams comprising the steps of placing particle-sizing instrumentation in a bitumen froth inlet stream to a gravity settling vessel subsequent to the addition of paraffinic solvent. The addition of solvent forming aggregates. A representative particle size distribution of entrained aggregates is measured with the particle-sizing instrumentation. The settling rate of the aggregates from the particle size distribution is determined mathematically. Operating data is collected while repeating one or more times the foregoing process steps and while changing one or more process conditions of the gravity-based process. A set of operating conditions are then established based upon particle size and settling rate from the generated data. The paraffinic solvent, gravity-based process is operated by adjusting process conditions to optimize the settling rate of the aggregates based upon the established set of operating conditions.

Diluted Bitumen Product

Bitumen Froth

Purge Gas

Solvent

Particle-sizing Instrumentation

Waste

Solvent

Tailings
FIG. 3

FIG. 4
FIG. 5

FIG. 6
FIG. 7
METHOD OF REMOVING SOLIDS FROM BITUMEN FROTH

CROSS REFERENCE TO RELATED APPLICATIONS

[0001] This application claims the benefit of U.S. Provisional Application No. 61/066,183, filed Feb. 19, 2008. This application is related to U.S. Provisional Application No. 61/065,371, entitled “Upgrading Bitumen in a Paraffinic Froth Treatment Process”, filed Feb. 11, 2008.

FIELD OF INVENTION

[0002] This invention relates generally to the removal of asphaltenes, other solids and water from bitumen froth that is generated in the processing of mined oil sands in producing hydrocarbon feed stocks suitable for refining.

BACKGROUND OF THE INVENTION

[0003] In the processing of mined oil sands, bitumen froth is generated through a combination of water/aqueous extraction, air flotation, and deaeration processes. Typically this deaerated bitumen froth takes the form of a bitumen emulsion containing approximately 60 wt % bitumen, 30 wt % water, and 10 wt % mineral solids (e.g., sand, clay).

[0004] The contaminants, such as water (carrying corrosive metal chlorides) and mineral solids in the bitumen froth have to be removed before the bitumen can be further upgraded or refined. For example, the total water and solids content (bottom solids and water analyzed by centrifugation) has to be less than about 0.5 vol. % in order to meet typical specifications for being transported by pipeline. Furthermore, the bitumen must contain only minimal amounts of impurities (e.g., less than a few hundred parts per million of ash product, the metal-containing compounds) if it is to be used as a feedstock for high-liquid conversion hydrocracking processes.

[0005] Traditionally, the solids and water have been removed from the bitumen froth using a naphthenic process. In the naphthenic process naphtha is added to the bitumen froth to reduce its density and viscosity. A series of inclined plate separators and centrifuges are then utilized to remove the bulk of the water and solids. This process typically results in a diluted bitumen product containing up to 3.5 vol. % solids and water. This bitumen product would likely require additional upgrading before it could be pipelined or used as a refinery feed stock.

[0006] Recently, a paraffinic process has been proposed as a more effective means to remove the solids and water from bitumen froth. In the paraffinic-froth treatment (PFT) process, a light aliphatic hydrocarbon solvent (typically pentane, hexane, or pentane/hexane blends) is blended with the bitumen froth. It has been shown that if the solvent concentration exceeds a certain critical level (the level depending on the solvent) certain components of the hydrocarbon (bitumen) known as asphaltenes will precipitate out of solution. Further experimental observations have shown that these asphaltenes, together with water droplets and solids dispersed in the bitumen, form aggregates as long as the onset conditions for asphaltene precipitation are satisfied. The resulting aggregates (or flocs) can be readily removed from the bitumen froth in gravity-based settling processes without the need for centrifuges or inclined-plate separators. This gravity-based settling process can generate a bitumen product containing less than 0.5 vol. % solids and water, meeting typical hydrocarbon pipeline specifications.

[0007] The primary criteria to assess product quality after treatment of the bitumen froth are water and solids content in the solvent-diluted bitumen. The water content of batch samples is often analyzed using Karl Fischer titration and solids content is often analyzed as ash content or filterable solids using standard procedures (see for example ASTM D473 and D4928). These procedures are time and labor-intensive and generally do not lend themselves to automatic on-stream implementation or permit timely adjustments to the operating conditions. Therefore, direct, real-time monitoring and control of the settling process in paraffinic froth treatment is not achieved with existing instrumentation and techniques.

[0008] In particular, Canadian Pat. No. 2,350,001 (the ‘001 reference) discloses a process controlled by monitoring the height of an interface (hydrocarbon/water) within a process vessel and adjusting flow rates of the vessel’s intake and withdrawal streams based on the interface height. The ‘001 reference fails to disclose the use of particle size distribution. Further, such an approach is undesirable because it requires long periods of time (e.g., hours) to respond to changes in the feed composition and changes in the level of the vessel may relate to parameters other than product quality and particle size.

[0009] U.S. Pat. App. No. 2004/0084623 (the ‘623 reference) discloses the use of near-infrared spectroscopy and chemometrics to determine asphaltene content, solvent-to-bitumen ratio, and density of a bitumen-soil stream. The ‘623 reference fails to disclose the determination or use of particle-size distribution to control a bitumen stream composition. Further, the method of the ‘623 reference is undesirable because it requires the development of a database of “calibration models,” which would require significant time and effort.

[0010] An article by Kurt Leschonski titled “Online Measurement of Particle Size Distributions in Gases and Liquids” from 1974 (the Leschonski reference) discloses a survey of technologies available for measuring particle-size distribution (PSD) and mentions the appropriateness of rapid response. The Leschonski reference does not disclose the use of on-line, real-time optical measurements. In fact, Leschonski teaches away from such an approach by listing “image analysis” of PSD as not appropriate for on-line analysis. See e.g., Table 1, item 4.

[0011] One current practice is to collect liquid samples from the product stream and perform standard analytical tests to determine water, mineral solids, or asphaltene content. These analyses take hours to complete, so the data are not available to make real-time process control decisions. Extensive measures must be employed to ensure the sample is representative and stable and care must be taken to address the safety concerns associated with the sampling from a pressurized feed stream containing hydrocarbon fluids. Furthermore, this type of data only reveals whether or not product specifications were being met at the time of sampling. The results do not provide much information regarding the gravity-based settling process nor do they indicate how close to the edge of an operating envelope one may be operating.

SUMMARY OF THE INVENTION

[0012] The described invention relates to a paraffinic solvent, gravity-based process for removing solids content from
bitumen froth streams comprising the steps of: a) providing particle-sizing instrumentation in a bitumen froth inlet stream to a gravity settling vessel subsequent to the addition of the paraffinic solvent, the solvent addition forming aggregates; b) measuring a representative particle size distribution of entrained aggregates with said instrumentation; c) determining mathematically the settling rate of the aggregates from said particle size distribution; d) collecting operating data while repeating one or more times steps a)-c) after changing one, or a combination of two or more, of the process conditions of the gravity-based process; e) establishing a set of desired process operating conditions based upon particle size and settling rate from the data generated in step d); and, f) operating said paraffinic solvent, gravity-based process for aggregate removal by adjusting process operating conditions to optimize the settling rate based upon the set of desired operating conditions established in step d).

[0013] In one or more preferred embodiments the process is preceded wherein an initial start-up process precedes the process of claim 1, said start-up process comprising: 1) isolating the particle-sizing instrumentation from the bitumen process stream, 2) then introducing hot process gas to the particle-sizing instrumentation for purging, 3) withdrawing the process gas after purging but without passing into the bitumen froth stream, 4) continuing said purging and withdrawing until process temperatures are reached, 5) and then reopening the flow of the bitumen froth stream through the particle-sizing instrumentation.

[0014] In further embodiments, the exposed portion of the instrumentation in the inlet stream is coated with an anti-fouling agent prior to the introduction of the bitumen froth in said inlet stream in a) of paragraph 0008 above. Additionally, it is preferred that in situ cleanings be conducted in the manner of the start-up options, with hot process gas or without, as described further below.

[0015] Thus the invention provides a method/methodology that would enable the measurement and control of the settling rate via particle size distribution (PSD) measurement associated with the gravity settling of asphaltene aggregates and solids in bitumen froth-paraffinic solvent process systems on-line. This method provides a unique process control tool that enables one skilled in the art to maintain the stringent product quality specifications.

BRIEF DESCRIPTION OF THE DRAWINGS

[0016] The present invention and its advantages will be better understood by referring to the following detailed description and the attached drawings in which:

[0017] FIG. 1 is a schematic for a paraffinic froth treatment process embodiment according to the invention where particle-sizing instrumentation is installed as a loop off of the bitumen froth feed stream after the addition of the paraffinic solvent.

[0018] FIG. 2 is a graphic illustration of the measured increase in median particle size with both increasing temperature and increasing solvent-to-bitumen ratio.

[0019] FIG. 3 is a graphic representation of the calculated increase in hindered settling rate of a median particle (as determined from the particle size distribution) with increasing temperature and solvent-to-bitumen ratio.

[0020] FIG. 4 is a graphic illustration of the measured particle size distribution in terms of the frequency of occurrence (%) of particles having chord lengths between 0 and 1000 microns when measured through a clean particle sizing instrument window.

[0021] FIG. 5 is a graphic illustration of the measured particle size distribution in terms of the frequency of occurrence (%) of particles having chord lengths between 0 and 1000 microns when measured through a fouled particle sizing instrument window.

[0022] FIG. 6 is a graphic representation of the measured particle size distribution in terms of the frequency of occurrence (%) of particles having chord lengths between 0 and 1000 microns when measured, first, before fouling, second, after fouling, and third, after in-situ cleaning.

[0023] FIG. 7 is a graphic illustration of median particle size over time predicted from a model based on earlier measurements and correlated to the temperature and solvent-to-bitumen ratio for actual running (solid line) and the actual median particle size as measured in situ over the same time period (large dots).

[0024] The invention will be described in connection with its preferred embodiments. However, to the extent that the following detailed description is specific to a particular embodiment or a particular use of the invention, this is intended to be illustrative only, and is not to be construed as limiting the scope of the invention. On the contrary, it is intended to cover all alternatives, modifications and equivalents that may be included within the scope of the invention, as defined by the appended claims.

DETAILED DESCRIPTION OF PREFERRED EMBODIMENTS

[0025] The process of the present invention relates to where real-time measurement of particle-size distribution in a process stream is used to make process control decisions in a gravity-based settling process. The process is particularly suited for the high-temperature paraffinic froth treatment process proposed for use in bitumen oil sand developments.

[0026] Particle-sizing instrumentation ("PSI") is placed in the inlet stream to the process, downstream of, or subsequent to, the point where paraffinic solvent is introduced to the bitumen froth. The particles (asphaltene flocs composed of water, mineral solids, precipitated asphaltenes, solvent, and sometimes, some bitumen) formed when the solvent is introduced to the froth are sized by the instrumentation prior to entering the settling vessel. Typical instrumentation may comprise a probe or a sample window, or equivalent, in the inlet stream, or more preferably, in an auxiliary loop, see FIG. 1, where at least a portion of the inlet stream, preferably all, is diverted for measurement by passing by the probe or window, before being returned to the inlet stream.

[0027] The standard paraffinic bitumen froth settling process comprises mixing a bitumen froth stream with paraffinic solvent which preferentially dissolves at least a large portion of the bitumen in the stream. The then combined stream of bitumen, water, solvent and entrained solids is conducted to a settling unit where the solvent and much of the bitumen rise to the top and are extracted in an overhead stream while much of the water and the entrained solids are withdrawn from the bottom of the unit. The process temperatures used in the invention processes are typically in excess of 50°C, but less than or equal to about 100°C, more typically in a range of from 60°C to 100°C. Though temperatures lower than 50°C can be used, the amount of solvent used must be increased to achieve the desired amount of asphaltene
precipitation. The operating pressures of the bitumen froth stream and in the settling unit are typically from about 100 to 150 psi (689.5 to 1,034 kPa); however, the embodiments described in this patent are not limited to these pressures.

[0028] The settling of particles (including aggregates) in a settling unit may occur under hindered or unhindered conditions, depending on the concentration and sizes of particles in the settling unit. Under free settling conditions, the particles settle independently and in unhindered fashion, with the settling rate mainly dependent on particle size. Under hindered settling conditions, the particles are sufficiently close to one another to restrict or hinder passage or egress of water and solvent from between particles. The particle settling rate decreases with increasing concentration and size of solid particles, and the settling rate is retarded by particle interference. The settling rate used in the practice of the present invention can be either an unhindered settling rate or a hindered settling rate, depending on conditions in the settling unit. Since hindered settling conditions are applicable to most settling units of paraffinic solvent, gravity-based processes for removing solids content from bitumen froth streams, the present disclosure discusses use of hindered settling rates. However, persons skilled in the art having benefit of this disclosure will recognize that an unhindered settling rate could be used if the particles in the settling unit do not experience significant hindered settling conditions. Persons skilled in the art would know how to mathematically calculate unhindered settling rates.

[0029] A preferred method for measuring a representative particle size distribution and determining mathematically from that a hindered settling rate is based on using a chord-length distribution (CLD) (or a suitable ensemble average of multiple distributions) of the particles. When the CLD is recorded by the PSI, it can be converted to a volume-based distribution of particle sizes using algebraic relationships well known in the art. Generally speaking, an ensemble average, a moving average (preferred), or other statistic measurement based upon the individual CLD’s will give better results than a single instantaneous statistic. Alternatively, more sophisticated models can be applied such as that offered by J. Wolfischke, T. Hocker, M. Mazzotti, “Restonion of PSD from Chord Length Distribution Data Using the Method of Projections onto Convex Sets”, Particle & Particle Systems Characterization, 22(2):81-98, 2005. Additional variables such as focal length can also be considered using an approach like that used by J. Wolfischke and M. Mazzotti, “Choice of the Focal Point Position Using Lasentec FBRM”, Particle & Particle Systems Characterization, 20(1):12-17, 2003.


[0032] Parametric studies can be used to correlate the observed aggregate size, and therefore settling rate, with a desired set of process operating conditions such as solvent/bitumen mass ratio (S:B), flowrate, pressure, and temperature. Operators may then adjust one, or a combination of two or more, of the process conditions (e.g., S:B, flowrate, pressure, and temperature) to affect the desired change in settling rate of the system. From this data, a parametric set of operating conditions can be established based upon particle size and settling rate.

[0033] Characteristic particle-size distribution patterns have been identified which signify that the optical window of the PSI has become partially or wholly obscured or coated with process fluid. Typically under optimal operating conditions the particle count, e.g., number of particles recorded by the PSI per second, will be a smooth curve in an exemplary range from about 2,000 to 6,000 particles per second, typically about 3,500, plus or minus from about 1,500 to 2,500. When the particle count begins to show spikes, especially large spikes, in the number of particles being counted, for example above about 10,000, or especially if above about 15,000 particles per second, up to as much as 40,000, or more, then the optical window is likely being obscured by adhering particles.

[0034] Start-up procedures have been developed which minimize the tendency of the process fluid to adhere to and to obscure the instrument’s optical window. Furthermore, an in-situ cleaning process has been developed to remove this obscuring layer. A combination of aromatic solvent (e.g., toluene or mixed aromatic solvents) washes and process gas (e.g., nitrogen, methane, helium, argon, natural gas or its component gases, etc.) purges of the piping containing the instrument has been shown to be effective in cleaning the probe window. See the further description below.

[0035] In a particularly preferred embodiment, an anti-fouling compound may be used as a coating to the exposed portion of the instrumentation, or its window, which reduces the potential frequency and severity of fouling. Furthermore, a coated window is easier to clean using the in situ process than an uncoated window.

[0036] Turning now to FIG. 1, particle-sizing instrumentation 12, preferably a Lasentec FBRM by Mettler-Toledo that uses a focused-beam reflectance measurement technique, in a particle distribution sizing process loop 20, is placed in the inlet stream 1 before a first settling vessel 2. This particle distribution sizing process loop may be much smaller than
depicted, and may treat only a representative sampling of the bitumen process stream, up to the entire stream in smaller operations. Placement in the inlet piping helps to assure that a representative sample of the bitumen froth has been obtained. In the first Froth Settling Unit 2, the heavier, larger aggregates settle and are taken out in a concentrated stream 4. This stream is then again diluted with solvent by solvent stream 5 and enters a second Froth Settling Unit 6 where the heaviest particles and water are removed as a tailings stream from the bottom. A thus increased-solvent stream is taken off as stream 7 and introduced into the inlet stream 1 at a point well before it enters the particle size distribution process loop 20 comprising the particle-size distribution instrument 12 and particle size distribution process streams 8 and 13. The new solvent-containing bitumen froth stream 1 can be diverted, for instance by closing of a gate valve V7 (though gate valves are discussed, any effective open/shut-off valve would be suitable as well), through an open gate valve V5, and becomes particle-sizing instrumentation process stream 8. Means of introducing a purge process gas stream 9 is provided by a gate valve V1 and additional means of introducing a solvent stream 10 is provided by a gate valve V2. A drain is typically located at a low point in the loop 20, stream 11, which can be isolated by a gate valve V3. The particle-sizing instrumentation (PSI) 12 then operates on the provided process stream 8. The instrument measured process stream 13 is then led and introduced back into the bitumen stream 1, for example through a gate valve V6 before entering the first Froth Settling Unit 2 (FSU). A waste stream 14 taken off from stream 13 is provided which can be opened or closed with a valve V4, this being used typically for purging or to adjust the pressure in the loop.

[0037] As noted above, an anti-fouling coating, such as dichlorodimethylsilane, may be applied to the exposed portion of the particle-sizing instrumentation 12, or window, in the inlet stream prior to the introduction of the bitumen froth to reduce the frequency and severity of fouling. Such windows are typically made of hard mineral substances that can transmit long wavelength light, for example, red laser sourced light. The instrumentation used in these examples was constructed of sapphire material. The coated window was observed to be easier to clean than an uncoated window using the in situ process. This coating can be applied by initially cleaning a probe’s optical window, for example, with toluene (or any aromatic solvent, or mix) and alcohol washes and then immersing the probe in a 10 vol % solution of the coating material, e.g., dichlorodimethylsilane dissolved in toluene, for 15 minutes or more. The probe is then removed and allowed to air dry. The immersion/air dry cycle may be repeated one or more times if a thicker coating is desired. Other suitable anti-fouling coating compounds include known coating materials that are stable and transparent under the operating conditions of the PFT process, e.g., TEFLEX®, and others.

[0038] After coating, if the preferred method is used, the particle-sizing instrumentation is exposed to a stream containing the bitumen froth, upstream of the first vessel, e.g., Froth Settling Unit 2 (FSU), but downstream of the point where the solvent stream 7 is introduced to the bitumen froth stream 1.

[0039] A preferred start-up procedure preceding the general process steps can be followed. Said start-up process can comprise: 1) isolating the PSI 12 from the bitumen process stream [opening V7 and closing V5]; 2) then introducing hot process gas as stream 9 to the PSI 12 [via V1] for purging; 3) withdrawing the process gas after purging as stream 14 [via V4] but without passing into the bitumen froth stream 1; 4) continuing said purging and withdrawing until process temperatures are reached; 5) and then reopening the flow of the bitumen froth stream through the PSI [opening V5 and V6 and closing V7]. The use of hot process gas is a preferred embodiment. Or more specifically, referring to FIG. 1: Starting with V7 open and V1, V2, V3, V4, V5 and V6 closed. Open V4.

[0040] Open V1 allowing hot process gas to flow through instrument loop 20 and PSI and discharge the gas stream to waste. Gas flow is continued until the PSI has reached process temperature. The duration of this step may be specified as a minimum time or based upon temperature instrumentation or in the PSI. Once process temperature is reached, V4 is closed. Confirm instrument loop pressure matches or exceeds line pressure.

Close V1.
Open V5.
Open V6.
Close V7.

[0041] If hot process gas is not available, the start-up process may comprise the following: (1) isolating the PSI 12 from the bitumen froth stream; (2) opening a waste gas withdrawal valve located in the PSI loop; (3) heating the PSI by applying heat to said instrumentation and at least a portion of connecting fittings and piping until process temperatures are reached; (4) increasing pressure in the PSI loop by closing said withdrawal valve and opening a process valve in said loop; (5) and then reopening the flow of the bitumen froth stream through the PSI 12. Or, more specifically referring to FIG. 1:

Start with V7 open and V1, V2, V3, V4, V5 and V6 closed.

Open V4.

[0042] Bring the PSI to process temperature by applying heat by means of steam lance, heat tracing, or other suitable heat source(s). The duration of this step may be specified as a minimum time or based upon temperature instrumentation or in the PSI. Once the PSI has reached process temperature, close V4. Open V1 allowing process gas to pressurize instrument loop. Confirm instrument loop pressure matches or exceeds line 1 pressure.

Close V1.
Open V5.
Open V6.
Close V7.

[0043] The described start-up procedures were developed to minimize the tendency of the process fluid to adhere to and to obscure the PSI’s optical window. Pre-heating of the piping and instrument to process temperature by circulating heated gas, external application of steam, heat tracing, or other heat source and pressurizing the instrument loop with dry gas equilibrates the instrument loop with the system before the PFT process fluids are introduced. In this way, when process fluid is introduced, there is minimal change in temperature or pressure and solvent vapor will not condense as droplets on the probe’s optical window.
A characteristic PSD was identified which signified that the probe window had been at least partially obscured by fouling. As noted above, particle counts increasing above about 10,000 counts/second, especially above 15,000 counts/second, were indicative of the fouling. This was repeatedly confirmed when the probe was removed from the process piping and a sticky, black film was observed on the probe's optical window.

In situ cleaning of the probe's window can be conducted without disrupting the process, when it has been determined that the probe's optical window has become obscured, see, for example, FIG. 5. This procedure can be completed as often as needed to keep the probe's window clean and measurements accurate. Referring again to FIG. 1, a PSI cleaning process follows, that process comprising: (1) isolating the PSI loop 20 from the bitumen froth stream 1; (2) opening a waste gas withdrawal valve V4 located in the PSI loop 20; (3) introducing a solvent stream 10 to the PSI 12 and (4) withdrawing the solvent as stream 14 after passing through the PSI 12; (5) monitoring the particle size distribution statistics in the PSI; (6) continuing steps (1) through (5) until the observed particle count is less than 100 counts/sec; (7) then stopping the introducing and the withdrawing of solvent (steps 3 and 4); (8) draining any accumulated solvent in the PSI 12; (9) flushing with process gas, and adding heat and any pressure needed with said gas to match operating conditions; (10) stopping the introduction and withdrawal of process gas; and (11) then re-opening the flow of the bitumen froth process stream to the PSI 12. More specifically, the process can be conducted as follows:

Open V7.
Close V6 and V5.

Open V4 to vent the instrumentation loop.

Open V2.

Pump solvent through the PSI 12 and the solvent exiting as waste stream 14. Monitor PSD statistics. Continue flowing solvent until observed particle count is less than 100 counts/sec, preferably less than 10 counts/sec. Typically, satisfactory results were obtained in 15 minutes or less.

Once particle count has been reduced, close V2.

Close V4.

Open V3 and allow remaining solvent in instrumentation loop to drain.

Open V1.

Blow out remaining solvent through drain (V3).

Follow start-up procedures described above (paragraphs [0023] or [0029]).

In an alternative embodiment, not shown, the PSI loop 20 can be installed in the dilute bitumen product line 3, instead of line 1 as illustrated. Thus valves V1, V2, V3, V4, V5, V6, V7 and streams 8, 9, 10, 11, 12, 13, 14, with the PSI 12, would be placed in product stream 3 after FSU1 2. The process would be operated in the manner described in this patent and would provide advantages of lesser fouling of the PSI and less need for in situ cleaning of the PSI probe's window.

EXAMPLES

Example 1

This example 1 demonstrates variation in median particle size and hindered settling rate with changes in the solvent-to-bitumen ratio (hereinafter “S:B”) or temperature.

A parametric study was conducted in a 15 barrel per day (bitumen froth feed) pilot plant. Commercial bitumen froth obtained from Syncrude Canada Ltd. (nominally 60 wt % bitumen, 30 wt % water, 10 wt % mineral solids) was fed to a paraffinic froth treatment (“PFT”) process using a 60/40 vol % blend of iso/normal pentane as the paraffinic solvent.

PSI 12 (Lasentec FBRM by Mettler-Toledo) was placed in the feed piping to the first froth settling unit (FSU1) 2 as indicated in FIG. 1. This location is downstream of the point where solvent in stream 7 is introduced into the bitumen froth feed 1 but upstream of the FSU1 2. A dichloromethylsilane coating had been applied to the probe's window (using the process described in paragraph [0030]) prior to insertion into the process piping to reduce the propensity for fouling. Initial baseline readings of the clean PSI indicated 84 counts/second. Process fluid was introduced into the PSI piping using the preferred start-up procedure described above in paragraph [0028] with hot nitrogen as the process gas.

The process was maintained at a number of predetermined set points (temperature and S:B) for a minimum of 5 minutes each, preferably 20 minutes or more during which time chord length distributions (CLD's) were recorded, once every 5 seconds. Data were stored on a computer for later analysis. During the study the S:B ratio was varied from 1.4 to 1.8 and the temperature was varied from 60 to 80°C.

Particle size distributions (PSDs) and statistics which included median, average, and particle count were monitored real-time during the parametric study. If fouling of the probe window was indicated (as identified using the procedure described in paragraph [0022]), the cleaning procedure described above was completed before the study was resumed. Typically, the probe operated for 2 or more hours before becoming fouled and requiring cleaning.

The PSDs collected at each process set point (or a subset) were ensemble averaged and statistics were computed. Three curves representing the median particle size in microns as a function of S:B ratio are shown in FIG. 2, representing temperatures of 60, 70, and 80°C, respectively.

This median particle size was used to compute the hindered settling rate, Uh, of the median particle at each process condition (temperature and S:B) using the Richardso-Zaki correlation (see paragraph 0020) given by the equation

\[ u_h = \frac{n(1 - \phi)^2}{\frac{4}{3} \pi d^3} \]

where,

\[ u_h = \frac{n(1 - \phi)^2}{\frac{4}{3} \pi d^3} \]

\[ u = \sqrt{\frac{4g(d_p - \mu)}{3n C_D}} \]

\[ Re = \frac{\mu_k h_d}{\rho} \]

\[ Re \]

\[ C_D = \begin{cases} \frac{24}{Re} & \text{Re} < 0.1 \\ \frac{24}{Re} \left(1 + 0.14 \text{Re}^{0.17}\right) & 0.1 < \text{Re} < 1,000 \\ \frac{24}{Re} \left(1 + 0.14 \text{Re}^{0.17}\right) & \text{Re} > 1,000 \end{cases} \]

ξ is the median particle size in meters, and the numerical values are given in Table 1. These computed hindered settling rates are shown in FIG. 3.
This curve can be used to establish operating guidelines, specifically the maximum flux rate or throughput, of the FSU 1 (see FIG. 1). For example, from FIG. 3 where S:B is 1.6 and the temperature is 70° C., we would expect the median particle size to settle at a rate within the range from about 1,400 to 1,600 mm/min. In this case, operation of the pilot plant was constrained so that the flux rate within the vessel would not exceed 1,400 mm/min to prevent the particles from being carried into the product stream. We can also determine that if temperature were increased to 80° C. the maximum flux rate could also be increased to about 1,800 mm/min, increasing the throughput of the process. Alternatively, this same effect could be obtained without increasing the temperature by increasing the S:B from 1.6 to approximately 1.75.

Example 2

This Example demonstrates the characteristic PSD associated with a fouled or obscured optical window.

A parametric study was conducted as in Example 1 including PSI placement, probe window coating and start-up procedure.

When the process fluid was first introduced past a clean PSI, PSDs similar to that shown in FIG. 4 were observed. In this case, 3,736 chords/second were recorded by the probe. However, after 140 min of operation, a PSD like that shown in FIG. 5 was observed. The high particle count, 15,831 chords/s, and noisy distribution are indicative of a fouled probe. The probe was removed from the instrumentation loop and examined. A thick, sticky, black film was observed, confirming that the probe surface was obscured.

Example 3

This Example demonstrates the effectiveness of the described cleaning process.

In this test, the PSI (Lasentec FBRM by Mettler-Toledo) was placed in the seed piping 1 to the first-stage settling vessel (FSU1) 2 as indicated in FIG. 1. As before, this location is downstream of the point where solvent in stream 7 is introduced into the bitumen froth feed but upstream of the FSU1 2. A dichlorodimethylsilane coating had been applied to the probe’s window as for Example 1 prior to insertion into the process piping to reduce the propensity for fouling. Initial baseline readings of the PSI indicated less than 100 counts. Process fluid was introduced into the PSI piping using the preferred start-up procedure described above for Example 1.

CLD’s similar to that shown in FIG. 6 were observed for the first approximately 90 minutes of operation (curve 20). After 90 minutes, however, a CLD characteristic of a fouled probe was observed (curve 21). At 99 minutes, the following cleaning procedure was completed. Referring to FIG. 1:

- Open V7.
- Close V6 and V5.
- Open V4 to vent the instrumentation loop.
- Open V2.

Pump solvent (toluene) through the PSI and into waste.

The CLD statistics were monitored and solvent was pumped until the observed particle count fell below 100 chords/sec. In this case it took 5 minutes of pumping toluene to adequately clean the window.

Close V2.
Close V4.
Open V3 and allow remaining solvent in instrumentation loop to drain.
Open V1.
Blow out remaining solvent through drain (V3).
The start-up procedures of paragraph [0028] were followed again.

Once the cleaning procedure was completed the observed CLD was substantially restored to its original distribution (curve 22) as shown in FIG. 6. This demonstrated that the cleaning procedure was effective in restoring the probe to its normal operation without the need to physically remove it from the instrument piping or disrupt the feed to the process.

Example 4

This Example demonstrates the potential for process control using the PSI and modeling based on its measurements.

This test was again conducted as in Example 1.

Data (CLDs) were collected with the PSI over a continuous 2-day period. The PSI was then computed using the procedures described previously at 4 hour intervals. The median of these PSDs distribution was determined and is shown in FIG. 7 (large dots).

The data collected in FIG. 3 were regressed to create an empirical model of the form:

\[ L = a + bT + cS + dS^2 + eT^2 + f \]

where \( L \) is the median particle size in microns, \( T \) is the temperature in °C., \( S \) is the solvent-to-bitumen ratio (dimensionless), and \( a, b, c, d, e, \) and \( f \) are adjustable parameters determined through least-squares regression. (Note alternate expressions could be used for the regression incorporating more or fewer independent variables.)

The temperature and solvent-to-bitumen ratio in the pilot period were also recorded during this same time period. These values (instantaneous values) of temperature and solvent-to-bitumen were used to predict the median particle size using the expression above. The resulting values are shown in FIG. 7 (black line). The reader can see that with minimal exception, the correlation obtained during the parametric study of Example 1 predicts the variation in median particle size as recorded by the PSI. In cases where significant deviation exists, the observed discrepancy is due to local (in time) variability in the composition of the bitumen froth fed to the system and does not represent an inherent weakness in the method.

TABLE 1

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Symbol</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Particle density</td>
<td>( p_e )</td>
<td>1.060 kg/m³</td>
</tr>
<tr>
<td>Liquid density</td>
<td>( p_l )</td>
<td>0.8 kg/m³</td>
</tr>
<tr>
<td>Particle volume fraction</td>
<td>( \Phi )</td>
<td>0.123</td>
</tr>
<tr>
<td>Richardson-Zaki exponent</td>
<td>( n )</td>
<td>5</td>
</tr>
<tr>
<td>Liquid viscosity</td>
<td>( \mu )</td>
<td>0.8 centipoise</td>
</tr>
<tr>
<td>Gravitational constant</td>
<td>( g )</td>
<td>9.81 m/s²</td>
</tr>
</tbody>
</table>
This expression (or one similar) could be implemented into a computer or other digital control system. If the current particle size in the process as measured by the PSI is undesirable, the appropriate temperature and/or solvent-to-bitumen ratio could be computed from the expression and the process set points could be changed. The PSI would then provide the necessary feedback to confirm that the process change had the desired influence on particle size. Such an approach does not require accurate knowledge of the composition of the bitumen froth since the real-time feedback and control allows the operator to maintain a desired particle size, and therefore settling rate, by changing temperature and/or solvent-to-bitumen ratio. This demonstrates that the PSI and the procedures described above could be implemented in a control system.

While the present invention may be susceptible to various modifications and alternative forms, the exemplary embodiments discussed above have been shown only by way of example. However, it should again be understood that the invention is not intended to be limited to the particular embodiments disclosed herein. Indeed, the present invention includes all alternatives, modifications, and equivalents falling within the true spirit and scope of the appended claims.

1. A paraffinic solvent, gravity-based process for removing solids content from bitumen froth streams comprising the steps of:
   a) placing particle-sizing instrumentation in a bitumen froth inlet stream to a gravity settling vessel subsequent to the addition of the paraffinic solvent, said solvent addition forming aggregates;
   b) measuring a representative particle size distribution of entrained aggregates with said instrumentation;
   c) determining mathematically the settling rate of said aggregates from said particle size distribution;
   d) collecting operating data while repeating one or more times the steps of a), b), and c) after changing one, or a combination of two or more, of a process condition of the gravity-based process;
   e) establishing a set of desired operating conditions based upon particle size and settling rate from the data generated in step d); and
   f) operating said paraffinic solvent, gravity-based process for aggregate removal by adjusting the process conditions to optimize the settling rate of the aggregates based upon the set of desired operating conditions established in step e).

2. The method of claim 1 wherein process step c) comprises (i) converting said particle size distribution to a volume-based distribution of particle sizes, and (ii) determining mathematically a hindered settling rate of said aggregates from said volume-based distribution of particle sizes.

3. The method of claim 2 wherein said instrumentation measures chord-length distribution in step b) and algebraic means are used to convert said chord-length distribution to a volume-based distribution of particle sizes in step c).

4. The method of claim 3 wherein the hindered settling rate of aggregates is determined from said volume-based distribution of aggregate sizes by application of the Richardson-Zaki correlation.

5. The method of claim 1 wherein the repeating of step e) one or more times is done after one or more cleaning process steps of aromatic solvent washes and cleaning gas purges of the instrumentation in the inlet stream have been conducted.

6. The method of claim 1 wherein the exposed portion of the instrumentation in the inlet stream is coated with an anti-fouling agent prior to the introduction of the bitumen froth in said inlet stream in step a).

7. The method of claim 1 wherein an initial start-up process precedes the process of claim 1, said start-up process comprising: 1) isolating the particle-sizing instrumentation from the bitumen process stream, 2) then introducing hot process gas to the particle-sizing instrumentation for purging, 3) withdrawing the process gas after purging but without passing into the bitumen froth stream, 4) continuing said purging and withdrawing until process temperatures are reached, and then 5) reopening the flow of the bitumen froth stream through the particle-sizing instrumentation.

8. The method of claim 1 wherein said particle-sizing instrumentation is in a particle-sizing instrumentation loop and wherein the paraffinic solvent, gravity-based process having an initial start up process, prior to steps a) of claim 1, comprising the steps of: 1) isolating the particle-sizing instrumentation loop from the bitumen froth stream, 2) opening a waste gas withdrawal valve located in particle-sizing instrumentation loop, 3) heating the particle-sizing instrumentation by applying heat to said instrumentation and at least a portion of connecting fittings and piping until process temperatures are reached, 4) increasing pressure in the particle-sizing instrumentation loop by closing said withdrawal valve and opening a process valve in said loop, and 5) then reopening the flow of the bitumen froth stream through the particle-sizing instrumentation.

9. The method of claim 4 additionally having a particle-sizing instrumentation cleaning process comprising: 1) isolating the particle-sizing instrumentation loop from the bitumen froth stream, 2) opening a waste gas withdrawal valve located in the particle-sizing instrumentation loop, 3) introducing a solvent stream to the particle-sizing instrumentation and withdrawing the solvent after passing through the particle-sizing instrumentation, 4) monitoring the particle size distribution statistics in the particle-sizing instrumentation, 5) continuing steps 1) through 4) until the observed particle count is less than 100 counts/sec, 6) then stopping the introducing and the withdrawing of solvent, 7) draining any accumulated solvent in the particle-sizing instrumentation, 8) flushing with process gas, and adding heat and any pressure needed with said gas to match operating conditions, 9) stopping the introduction and withdrawal of process gas, and 10) re-opening the flow of the bitumen froth process stream to the particle-sizing instrumentation.

10. The method of claim 1 wherein the particle-sizing instrumentation has an access window and said access window is coated with a transparent coating.

11. The method of claim 10 wherein said transparent coating comprises at least one of dichlorodimethylsilane or Teflon.

12. The method of claim 1 wherein in step a) said placing of the particle-sizing instrumentation is in the diluted bitumen product stream instead of the bitumen froth inlet stream.

13. The method of claim 1, wherein steps a), b), c), d), and e) are completed in real-time.

14. The method of claim 13, wherein steps a), b), c), d), and e) are completed in about 15 minutes.
15. The method of claim 1, wherein the process conditions are selected from the group consisting of: a process temperature, a process pressure, a process solvent to bitumen ratio, a flow rate in the gravity settling vessel, and any combination thereof.

16. The method of claim 15, wherein the process temperature is from about 50 degrees Celsius (°C.) to about 100°C.

17. The method of claim 15, wherein the process pressure is from about 100 pounds per square inch (psi) to about 150 psi.

18. The method of claim 15, wherein the solvent to bitumen ratio (S:B) is from about 1.4 to about 1.8.

19. The method of claim 15, wherein the flow rate in the gravity settling vessel is from about 1,000 millimeters per minute (mm/min) to about 2,500 mm/min.

20. The method of claim 1, wherein the particle size distribution is measured using Focused Beam Reflectance Measurements (FBRM).

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