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(54) Title: METHOD OF PROCESSING A BITUMINOUS FEED WITH FEEDBACK CONTROL

(57) Abstract: Described herein is a method of processing a bituminous feed. The bituminous feed is contacted with an extraction liquor to form a slurry. A bridging liquid is added to the slurry, and, solids are agitated within the slurry to form an agglomerated slurry comprising agglomerates and a low solids bitumen extract. In order to control agglomeration, the slurry is analyzed and the processing method is adjusted accordingly.



METHOD OF PROCESSING A BITUMINOUS FEED WITH FEEDBACK CONTROL**CROSS REFERENCE TO RELATED APPLICATIONS**

[0001] This application claims the benefit of Canadian patent application number
5 2,741,280 filed on May 27, 2011 entitled METHOD OF PROCESSING A BITUMINOUS FEED
WITH FEEDBACK CONTROL, the entirety of which is incorporated herein.

FIELD

[0002] The present disclosure relates generally to the field of hydrocarbon extraction
from mineable deposits, such as bitumen from oil sands.

10 **BACKGROUND**

[0003] Methodologies for extracting hydrocarbon from oil sands have required
energy intensive processing steps to separate solids from the products having commercial
value.

[0004] Solvent extraction processes for the recovery of the hydrocarbons have been
15 proposed as an alternative to water extraction of oil sands. However, the commercial
application of a solvent extraction process has, for various reasons, eluded the oil sands
industry. A major challenge to the application of solvent extraction to oil sands is the
tendency of fine particles within the oil sands to hamper the separation of solids from the
hydrocarbon extract. Solids agglomeration is a technique that can be used to deal with this
20 challenge.

[0005] Solids agglomeration is a size enlargement technique that can be applied
within a liquid suspension to assist solid-liquid separation. The process involves
agglomerating fine solids, which are difficult to separate from a liquid suspension, by the
addition of a second liquid. The second liquid preferentially wets the solids but is immiscible
25 with the suspension liquid. With the addition of an appropriate amount of the second liquid
and a suitable agitation, the second liquid displaces the suspension liquid on the surface of
the solids. As a result of interfacial forces between the three phases, the fines solids
consolidate into larger, compact agglomerates that are more readily separated from the
suspension liquid.

30 **[0006]** Solids agglomeration has been used in other applications to assist solid-liquid

separation. For example, the process has been used in the coal industry to recover fine coal particles from the waste streams produced during wet cleaning treatments (see for example, U.S. Patent Nos. 3,856,668 (Shubert); 4,153,419 (Clayfield); 4,209,301 (Nicol et al.); 4,415,445 (Hatem) and 4,726,810 (Ignasiak)). Solids agglomeration has also been
5 proposed for use in the solvent extraction of bitumen from oil sands. This application was coined Solvent Extraction Spherical Agglomeration (SESA). A more recent description of the SESA process can be found in Sparks et al., Fuel 1992 (71); pp 1349-1353.

[0007] Previously described methodologies for SESA have not been commercially adopted. In general, the SESA process involves mixing oil sands with a hydrocarbon
10 solvent, adding a bridging liquid to the oil sands slurry, agitating the mixture in a slow and controlled manner to nucleate particles, and continuing such agitation to permit these nucleated particles to form larger multi-particle spherical agglomerates for removal. The bridging liquid is preferably water or an aqueous solution since the solids of oil sands are mostly hydrophilic and water is immiscible with hydrocarbon solvents.

[0008] The SESA process described by Meadus et al. in U.S. Patent No. 4,057,486, involves combining solvent extraction with solids agglomeration to achieve dry tailings suitable for direct mine refill. In the process, organic material is separated from oil sands by mixing the oil sands material with an organic solvent to form a slurry, after which an aqueous
15 bridging liquid is added in the amount of 8 to 50 wt% of the feed mixture. By using controlled agitation, solid particles from oil sands come into contact with the aqueous bridging liquid and adhere to each other to form macro-agglomerates of a mean diameter of 2 mm or greater. The formed agglomerates are more easily separated from the organic solvent compared to un-agglomerated solids. This process permitted a significant decrease in water use, as compared with conventional water-based extraction processes. The multi-phase
20 mixture need only be agitated severely enough and for sufficient time to intimately contact the aqueous liquid with the fine solids. The patent discloses that it is preferable that the type of agitation be a rolling or tumbling motion for at least the final stages of agglomeration. These types of motion should assist in forming compact and spherical agglomerates from which most of the hydrocarbons are excluded. The formed agglomerates are referred to as
25 macro-agglomerates because they result from the consolidation of both the fine particles (sized less than 44 μm) and the coarse particles (sized greater than 200 μm) found in the oil sands.
30

[0009] U.S. Patent No. 3,984,287 (Meadus et al.) and U.S. Patent No. 4,406,788 (Meadus et al.) both describe apparatuses for extracting bitumen from oil sands while

forming macro-agglomerates for easy solid-liquid separation. U.S. Patent No. 3,984,287 (Meadus et al.) describes a two vessel agglomeration apparatus. The apparatus comprises a mixing vessel for agitating the oil sands, the bridging liquid, and the solvent to form a slurry with suspended agglomerates. The slurry is screened in order to remove a portion of the hydrocarbon liquid within which the bitumen product is dissolved. The agglomerates are then directed to a tapered rotating drum where they are mixed with additional solvent and bridging liquid. The additional solvent acts to wash the excess bitumen from the agglomerates. The additional bridging liquid allows the agglomerates to grow by a layering mechanism and under the increasing compressive forces produced by the tapered rotating drum bed depth. The compressive forces act to preferentially remove hydrocarbon liquid from the pores of the agglomerates such that, when optimal operating conditions are imposed, the pores of the agglomerates end up being filled with only the bridging liquid, and the solvent that remains on the surface of the agglomerates is easily recovered. U.S. Patent No. 4,406,788 (Meadus et al.) describes a similar apparatus to that of U.S. Patent No. 3,984,287 (Meadus et al.), but where the extraction and agglomeration processes occurs within a single vessel. Within this vessel, the flow of solvent is counter-current to the flow of agglomerates which results in greater extraction efficiency.

[0010] The above-mentioned patents describe methods of using the fines within oil sands and an aqueous bridging liquid to promote the consolidation of the coarse oil sands particles into compact macro-agglomerates having minimal entrained hydrocarbons and which are easily separated from the hydrocarbon liquid by simple screening. This macro-agglomeration process may be suitable for oil sands feeds comprising greater than 15 wt% fines. For oil sands with a lesser amount of fines, the resulting agglomerates show poor strength and a significant amount of hydrocarbons entrained within their pores. The inability of the macro-agglomeration process to produce agglomerates of similar solid-liquid separation characteristics regardless of oil sands feed grade, is a limitation. This limitation can be mitigated by using a water and fine particle slurry as the bridging liquid. U.S. Patent No. 3,984,287 (Meadus et al.) reveals that middlings of a primary separation vessel of a water-based extraction process or sludge from the water-based extraction tailings ponds may be used as the bridging liquids with high fines content. It has been shown that when sludge is used as the bridging liquid, the addition of the same amount of sludge per unit weight of oil sands feed may result in the production of agglomerates of the same drainage properties regardless of oil sands quality.

[0011] U.S. Patent No. 4,719,008 (Sparks et al.) describes a process to address the agglomeration challenge posed by varying ore grades by means of a micro-agglomeration

procedure in which the fine particles of the oil sands are consolidated to produce agglomerates with a similar particle size distribution to the coarser grained particles of the oil sands. Using this micro-agglomeration procedure, the solid-liquid separation behavior of the agglomerated oil sands will be similar regardless of ore grade. The micro-agglomeration process is described as occurring within a slowly rotating horizontal vessel. The conditions of the vessel favor the formation of large agglomerates; however, a light milling action is used to continuously break down the agglomerates. The micro-agglomerates are formed by obtaining an eventual equilibrium between cohesive and destructive forces. Since rapid agglomeration and large agglomerates can lead to bitumen recovery losses owing to entrapment of extracted bitumen within the agglomerated solids, the level of bridging liquid is kept as low as possible commensurate with achieving economically viable solid-liquid separation.

[0012] The micro-agglomeration process described in U.S. Patent No. 4, 719,008 (Sparks et al.) has several disadvantages that have thus far limited the application of the technology. Some of these disadvantages will now be described.

[0013] The micro-agglomeration process described in U.S. Patent No. 4,719,008 (Sparks et al.) requires careful control of the binding liquid to solids ratio. If the amount of bridging liquid added to the process is in excess of the required amount, rapid growth of agglomerates can lead to bitumen recovery losses owing to entrapment of bitumen within the agglomerated solids. However, if the amount of bridging liquid added to the process is too low, insufficient agglomeration increases the amount of dispersed fines in the liquid suspension which hampers solids-liquid separation. In U.S. Patent No. 4,719,008, a ratio between 0.112 and 0.12 was identified as an appropriate range for bridging liquid to solids ratio for a particular type of low grade ore. Maintaining the ratio within a narrow range during the actual field operation of the agglomeration process would be a challenge. Furthermore, the desired amount of bridging liquid for the agglomeration process will depend on the ore quality and the chemistry of the fines. Because the ore quality and chemistry will change on a frequent basis as different mine shelves are progressed, the recipe of the agglomeration process may need to change accordingly in order to maintain the agglomeration output within an acceptable range.

[0014] In previously described SESA processes, the bridging liquid is either added directly to the dry oil sands or is added to the oil sands slurry comprising the oil sands and the hydrocarbon solvent. In the former scenario, bitumen extraction and particle agglomeration occurs simultaneously. For this reason, the growth of agglomerates may

hamper the dissolution of the bitumen into the solvent, it may lead to trapping of bitumen within the agglomerates, and it may result in an overall increase in the required residence time for bitumen extraction. In the scenario where the bridging liquid is added to the oil sands slurry, excessive agglomeration may occur in the locations of bridging liquid injection.

5 These agglomerates will tend to be larger than the desired agglomerate size and result in an increase in the viscosity of the slurry. A higher slurry viscosity may hamper the mixing needed to uniformly distribute the bridging liquid throughout the remaining areas of the slurry. Poor bridging liquid dispersion may result in a large agglomerate size distribution, which is not preferred.

10 **[0015]** An important step in the agglomeration process is the distribution of the bridging liquid throughout the liquid suspension. Poor distribution of the bridging liquid may result in regions within the slurry of too low and too high bridging liquid concentrations. Regions of low bridging liquid concentrations may have no or poor agglomeration of fine solids, which may result in poor solid-liquid separation. Regions of high bridging liquid
15 concentration may have excess agglomeration of solids, which may result in the trapping of bitumen or bitumen extract within the large agglomerates. In the process described in U.S. Patent No. 4,719,008 (Sparks et al.), the milling action of the rotating vessel acts to both breakup large agglomerates and distribute the bridging liquid throughout the vessel in order to achieve uniform agglomerate formation. In a commercial application, the rotating vessel
20 would need to be large enough to process the high volumetric flow rates of oil sands. Accomplishing uniform mixing of the bridging liquid in such a large vessel would require a significant amount of mixing energy and long residence times.

[0016] Coal mining processes often produce aqueous slurries comprising fine coal particles. Solids agglomeration has been proposed as a method of recovering these fine
25 coal particles, which may constitute up to 30 wt.% of the mined coal. In the solids agglomeration process, the hydrophobic coal particles are agglomerated within the aqueous slurry by adding an oil phase as the bridging liquid. When the aqueous slurry, with bridging liquid, is agitated, the coal particles become wetted with an oil layer and adhere to each other to form agglomerates. The hydrophilic ash particles are not preferentially wetted by
30 the oil phase and, as a result, remain un-agglomerated and suspended in the aqueous phase. The agglomerated coal material, with reduced ash content, is readily separated from the aqueous slurry by mechanical methods such as screening.

[0017] U.S. Patent No. 4,153,419 (Clayfield et al.) describes a process for the agglomeration of coal fines within an aqueous slurry by staged addition of a bridging liquid to

the aqueous slurry. Each agglomeration stage comprises the addition of a bridging liquid to the slurry, agitation of the mixture, and removal of agglomerates from the aqueous slurry. The inventors found that performing the agglomeration process in at least two stages yielded higher agglomeration of the coal particles as compared to the case where the same amount of bridging liquid was added in one agglomeration stage.

[0018] U.S. Patent No. 4,415,445 (Van Hattem et al.) describes a process for the agglomeration of coal fines within an aqueous slurry by the addition of a bridging liquid and the addition of seed pellets that are substantially larger than the coal fines. The presence of seed pellets induces agglomerate growth to occur predominately by a layering mechanism rather than by a coalescence mechanism. Since the rate of agglomeration occurs much faster by layering compared to coalescence, the process described therein allows agglomerates to form very quickly so that, for a given residence time, a higher throughput of agglomerates can be obtained compared to the throughput obtainable in the absence of seed pellets.

[0019] U.S. Patent No. 4,726,810 (Ignasiak) describes a process for the agglomeration of coal fines within an aqueous slurry by the addition of a bridging liquid comprising a low quality oil, such as bitumen, and a light hydrocarbon diluent, such as kerosene. The aqueous slurry mixture is agitated by pumping it through a pipeline within which coal particles agglomerate and may later be separated from the slurry by screening. The process allows for the selective agglomeration of low-rank coal using substantially a low quality oil.

[0020] It would be desirable to provide an alternative or improved method for processing a bituminous feed.

SUMMARY

[0021] The present disclosure relates to a method of processing a bituminous feed. The bituminous feed is contacted with an extraction liquor to form a slurry. A bridging liquid is added to the slurry, and solids are agitated within the slurry to form an agglomerated slurry comprising agglomerates and a low solids bitumen extract. In order to control agglomeration, the slurry is analyzed and the processing method is adjusted accordingly.

[0022] In a first aspect, the present disclosure provides a method of processing a bituminous feed, the method comprising: a) contacting the bituminous feed with an extraction liquor to form a slurry, wherein the extraction liquor comprises a solvent; b) adding a bridging liquid to the slurry, and agitating solids within the slurry, to form an agglomerated

slurry comprising agglomerates and a low solids bitumen extract; c) measuring at least one property of the agglomerated slurry, the agglomerates, or the low solids bitumen extract; and d) comparing the at least one property to a target range, and where the at least one property that is measured does not fall within the target range, adjusting at least one parameter of the method of processing the bituminous feed, for controlling the agglomeration.

[0023] Other aspects and features of the present disclosure will become apparent to those ordinarily skilled in the art upon review of the following description of specific embodiments in conjunction with the accompanying figures.

BRIEF DESCRIPTION OF THE DRAWINGS

[0024] Embodiments of the present disclosure will now be described, by way of example only, with reference to the attached Figures.

[0025] Fig. 1 is a flow chart illustrating a disclosed embodiment.

[0026] Fig. 2 is a schematic illustrating a disclosed embodiment.

[0027] Fig. 3 is a schematic illustrating a disclosed embodiment.

[0028] Fig. 4 is a schematic illustrating a disclosed embodiment.

[0029] Fig. 5 is a schematic illustrating a disclosed embodiment.

[0030] Fig. 6 is a schematic illustrating a disclosed embodiment.

[0031] Fig. 7 is a calibration curve relating bitumen content of a bitumen extract comprised of bitumen and solvent to the measured density of the bitumen extract.

[0032] Fig. 8 is a graph of bitumen recovery and initial filtration rate as a function of extraction time with the agglomeration time kept constant at 2 minutes.

[0033] Fig. 9 is a graph of bitumen recovery and initial filtration rate as a function of agglomeration time with the extraction time kept constant at 5 minutes.

[0034] Fig. 10 is a schematic illustrating a disclosed embodiment.

DETAILED DESCRIPTION

[0035] The present disclosure relates to a method of processing a bituminous feed using feedback control. This method may be combined with aspects of other solvent

extraction processes, including, but not limited to, those described above in the background section, and those described in Canadian Patent Application Serial No. 2,724,806 ("Adeyinka et al."), filed December 10, 2010 and entitled "Processes and Systems for Solvent Extraction of Bitumen from Oil Sands".

- 5 **[0036]** Prior to describing embodiments specifically related to the feedback control, a summary of the processes described in Adeyinka et al. will now be provided.

10 **[0037]** **Summary of Processes of Solvent Extraction Described in Adeyinka et al.** To extract bitumen from oil sands in a manner that employs solvent, a solvent is combined with a bituminous feed derived from oil sand to form an initial slurry. Separation of the initial slurry into a fine solids stream and coarse solids stream may be followed by agglomeration of solids from the fine solids stream to form an agglomerated slurry. The agglomerated slurry can be separated into agglomerates and a low solids bitumen extract. Optionally, the coarse solids stream may be reintroduced and further extracted in the agglomerated slurry. A low solids bitumen extract can be separated from the agglomerated
15 slurry for further processing. Optionally, the mixing of a second solvent with the low solids bitumen extract to extract bitumen may take place, forming a solvent-bitumen low solids mixture, which can then be separated further into low grade and high grade bitumen extracts. Recovery of solvent from the low grade and/or high grade extracts is conducted, to produce bitumen products of commercial value.

20 **[0038]** As outlined in the summary section, and now with reference to Figure 1, the present disclosure relates to a method of processing a bituminous feed. The bituminous feed is contacted with an extraction liquor to form a slurry (**102**). A bridging liquid is added to the slurry and solids are agitated within the slurry to form an agglomerated slurry comprising agglomerates and a low solids bitumen extract (**104**). At least one property of the
25 agglomerated slurry, the agglomerates, or the low solids bitumen extract is measured (**106**). The at least one measured property is compared to a target range. Where the at least one measured property that is measured does not fall within the target range, at least one parameter of the method is adjusted, for controlling the agglomeration (**108**).

30 **[0039]** The term "bituminous feed" refers to a stream derived from oil sands that requires downstream processing in order to realize valuable bitumen products or fractions. The bituminous feed is one that comprises bitumen along with undesirable components. Such a bituminous feed may be derived directly from oil sands, and may be, for example raw oil sands ore. Further, the bituminous feed may be a feed that has already realized some initial processing but nevertheless requires further processing. Also, recycled streams that

comprise bitumen in combination with other components for removal as described herein can be included in the bituminous feed. A bituminous feed need not be derived directly from oil sands, but may arise from other processes. For example, a waste product from other extraction processes which comprises bitumen that would otherwise not have been recovered, may be used as a bituminous feed. Such a bituminous feed may be also derived directly from oil shale oil, bearing diatomite or oil saturated sandstones.

[0040] As used herein, "agglomerate" refers to conditions that produce a cluster, aggregate, collection or mass, such as nucleation, coalescence, layering, sticking, clumping, fusing and sintering, as examples.

[0041] **Figure 2** is a schematic of a method of processing a bituminous feed with additional steps including downstream solvent recovery. Feedback control is not illustrated in Figure 2. The extraction liquor (**202**) is mixed with a bituminous feed (**204**) from oil sands in a slurry system (**206**) to form a slurry (**208**). The extraction liquor comprises a solvent and is used to extract bitumen from the bituminous feed. The slurry is fed into an agglomerator (**210**). Extraction may begin when the extraction liquor (**202**) is contacted with the bituminous feed (**204**) and a portion of the extraction may occur in the agglomerator (**210**). A bridging liquid (**212**) is added to the agglomerator to assist agglomeration of the slurry. Agitation of the slurry is also used to assist agglomeration.

[0042] The agglomerated slurry (**214**), comprising agglomerates and a low solids bitumen extract, is sent to a solid-liquid separator (**216**) to produce a low solids bitumen extract (**218**) and agglomerates (**220**).

[0043] The following additional steps may also be performed. The low solids bitumen extract is sent to a solvent recovery unit (**222**) to recover solvent (**224**) leaving a bitumen product (**226**). The agglomerates (**220**) are sent to a tailings solvent recovery unit (**228**) to recover solvent (**230**) leaving dry tailings (**232**).

[0044] In one embodiment, the bituminous feed is dry oil sands, which is contacted with extraction liquor that free of bridging liquid in a slurry system to produce a pumpable slurry. The slurry may be well mixed in order to dissolve the bitumen. In this embodiment, the bitumen is first extracted from the bituminous feed prior to agglomeration in order to prevent (or limit) the agglomeration process from hampering the dissolution of bitumen into the extraction liquor. In another embodiment, the bridging liquid may be directly mixed with the bituminous feed before or at the same time as the extraction liquor so that bitumen extraction and agglomeration occur simultaneously. In this embodiment, the bridging liquid

is added before or at the same time as the extraction liquor in order to minimize the dispersion of fines, which may reduce the solids content of the bitumen extract after the agglomeration process.

5 **[0045]** In one embodiment, the formed agglomerates are sized on the order of 0.1-1.0 mm, or on the order of 0.1-0.3 mm. In one embodiment, at least 80 wt.% of the formed agglomerates are 0.1-1.0 mm or 0.1 to 0.3 mm in size. The rate of agglomeration may be controlled by a balance between intensity of agitation within the agglomeration vessel, shear within the vessel which can be adjusted by for example changing the shape or size of the vessel, fines content of the slurry, bridging liquid addition, and residence time of the
10 agglomeration process.

[0046] The agglomeration of the fines within the slurry plays an important role in the recovery of bitumen from the oil sands. Little or no agglomeration of the fines hampers solid-liquid separation since fine particles interfere with the filtration process and/or increase the solids content of the low solids bitumen extract. However, excess agglomeration of
15 solids results in entrapment of bitumen extract within the large agglomerates. Thus, it is desirable to control the agglomeration process with a view to achieving the desired agglomerates, such as agglomerates of a desired size, density, composition, other parameter, or a combination thereof.

[0047] The level of agglomeration will be affected by many factors among the most
20 consequential are the composition of the bituminous feed (for instance as a result of ore quality), the amount of bridging liquid added, the method by which the bridging liquid is added, the residence time of the extraction and agglomeration processes, the type and intensity of agitation, the shear environment, the amount of any additional solids that are added, and the surface chemistry of the fines. .

25 **[0048]** Because the ore quality, a measure of the ore chemistry and physical characteristics, may change on a very frequent basis as different mine shelves are progressed, the recipe for agglomeration may vary resulting in varying agglomeration. Thus, it is desirable to use a process that can be adjusted to account for feed variation and/or the resultant agglomeration outputs.

30 **[0049]** According to one embodiment, the bituminous feed and/or at least one of the agglomeration outputs (the agglomerates and the low solids bitumen extract) are analyzed as agglomeration proceeds. This information is then used to adjust the process, for instance by increasing or decreasing added solids content, adjusting the amount of bridging liquid

added, adjusting the residence time of the extraction and/or agglomeration processes, adjusting intensity of agitation, or adjusting the shear environment to seek more desired output(s). These parameters may be adjusted individually or in combination in order to maximize the effective response of the control system.

5 **[0050]** Figure 3 illustrates one embodiment, where the following steps are performed:

1. Measure (A) properties of the slurry (**302**) comprised of bituminous feed and extraction liquor. In another embodiment, the bituminous feed may be measured prior to contact with the extraction liquor.
- 10 2. Combine the slurry (**302**) with a bridging liquid (**304**) and add to an agglomerator (**306**).
3. Measure (B) properties of one or more outputs (**308**) (i.e. the agglomerates and the low solids bitumen extract) of the agglomeration process. The measurement may be performed continuously.
- 15 4. Use the measurements in a controls system (**310**) to adjust a parameter of the process. One option is to adjust the amount of bridging liquid (**304**) that is added to the slurry. Another option is to adjust the composition of the bridging liquid added to the slurry. Another option is to adjust the methods and locations of the bridging liquid addition in the process. Another option is to adjust the solid content of the slurry. Another option is to
20 adjust the intensity of agitation of the slurry. Another option is to adjust the residence time of the extraction process. Another option is to adjust the residence time of the agglomeration process. Yet another option is to adjust the shear environment of the agglomeration by changing for example the size or shape of the vessel.

[0051] Measurable properties of a bituminous feed which could be used include but
25 are not limited to: (i) fines content, (ii) moisture content, (iii) level of insoluble organics, (iv) quantity of bitumen present, (v) clays content, (vi) clay chemistry, (vii) particle size distribution, (viii) density, (ix) electrical properties such as conductivity. Standard tests are available for all of these measurements; for example methylene blue testing is a well known method that can be used to quantify the quantity of clays in the oil sands ore.

30 **[0052]** Measurable properties of the outputs of the solvent extraction with solids agglomeration process include but are not limited to: (i) particle size distribution of output solids, (ii) filtration rate of slurry, (iii) fines content of the low solids bitumen extract, (iv)

bitumen content of low solids bitumen extract, and (v) viscosity (rheology) of the slurry. The values of these properties are strongly impacted by the solvent extraction process and thus can be used in the control system described herein. Other measurable properties include: (vi) hydrocarbon content of the output solids, (vii) moisture content of agglomerates, (viii) attrition and/or strength of the agglomerated solids, (ix) electrical properties, and (x) yield strength of the slurry.

[0053] Particle Size Distribution Property. The particle size distribution of the output solids can be measured by integrating an on-line particle size measurement device such as a Retsch Technology Camizer. A slip stream can be taken from the slurry, filtered to remove liquid, and then measured to analyze particle size distribution. The particle size distribution of output agglomerates may have a measured D50 of between 100 microns and 300 microns, or the agglomerates might have a measured D50 of between 300 and 1000 microns, or the agglomerates might have a measured D50 of between 1000 and 2000 microns. It is preferable that the measured D50 be between 100 and 300 microns because such a particle size distribution would insure good solid-liquid separation rate while reducing the entrapment of bitumen extract within the pores of the agglomerates.

[0054] Filtration Rate Property. The filtration rate of the slurry can be measured by integrating an on-line filtration device with the pipeline. A slip stream can be taken from the slurry and the rate of filtration can be measured, or alternatively the filtration rate may be directly measured if a filtration process is included in the processing of the slurry in the solid-liquid separator. In the case of a slip stream filtration, the filter medium should be similar in material and pore size to that which is used in the solid-liquid separator. Exemplary filtration device include, but are not limited to, lab scale chamber presses and diaphragm filter presses. The filtration rate of the slurry is preferably in the range of 0.2 to 1 mL/cm²sec. Higher filtration rates may be suitable; however, care should be taken to ensure that such filtration rates are not due to excessive channeling.

[0055] Fines Content Property. The fines content of the low solids bitumen extract may be measured using several methods that are well known in the art. However, a method that quickly measures the solid content is preferable. Such a method may involve taking a slip stream of the slurry and filtering it to produce a low solids bitumen extract or directly sampling the low solids bitumen extract from the solid-liquid separator. The density of the bitumen extract and a micro-filtered bitumen extract is then measured. The bitumen extract can be filtered through a micro-filter with a nominal pore size of 0.45 microns. Suitable density measuring devices include vibration type liquid density meters. The difference in

density of the bitumen extract and micro-filtered bitumen extract can be correlated with solid content, S by using following equation :

$$S = \frac{\frac{1}{\rho_T} - \frac{1}{\rho_E}}{\frac{1}{\rho_S} - \frac{1}{\rho_E}}$$

where ρ_T is the measured density of the low solids bitumen extract and ρ_E is the measured
 5 density of the micro-filtered bitumen extract. ρ_S is the solid density that may be obtained by
 experimental calibration or approximated to have a value between 2.3 to 2.6 g/cm³. The
 solid content of the low solids bitumen extract is preferably less than 2 wt%, or preferably
 less than 1 wt%, or even more preferably less than 0.5 wt%.

[0056] Still another method of measuring the fines content may be an optical
 10 method, such as to dilute a low solids bitumen extract stream with excess solvent and then
 measure the turbidity of bitumen extract and micro-filtered bitumen extract. The difference in
 turbidity may be calibrated with fines content of the low solids bitumen extract.

[0057] Bitumen Content Property. During the extraction process as bitumen from
 the oil sands dissolves into the extraction liquor, the density of the low solids bitumen extract
 15 increases. The bitumen content of the low solids bitumen extract can be estimated by
 measuring the density of the low solids bitumen extract. A slip stream can be taken from the
 slurry and filtered to produce the low solids bitumen extract or the low solids bitumen extract
 can be sampled from the output of the solid-liquid separator. The low solids bitumen extract
 can be filtered through a micro-filter with a nominal pore size of 0.45 microns to obtain a
 20 solid-free bitumen extract. The density of the solid-free bitumen extract can be measured
 using an on-line density meter. The density of the bitumen extract can then be used to
 approximate the bitumen content of the solid-free bitumen extract. Figure 7 is a calibration
 curve relating bitumen content of a bitumen extract comprised of bitumen and solvent to the
 measured density of the bitumen extract. The measurement can also be used to determine
 25 the degree of bitumen extraction from the oil sands at different points along the extraction
 and agglomeration processes.

[0058] Viscosity Property. The particle size distribution of the oil sands slurry has a
 strong impact on the viscosity of the slurry. Slurries with a high fines content is expected to
 have a high viscosity. The slurry viscosity is expected to decrease as the average particle
 30 size of the slurry increases. Additionally, since a hydrocarbon phase is the continuous fluid

in the slurry, water chemistry will have much less of an impact on the viscosity/rheology behavior of the slurry compared to the impact water chemistry has on the viscosity/rheology of water-based extraction slurries. This fact makes correlation of particle size distribution with rheology much simpler for the oil sands slurries described herein. Thus, in one
5 embodiment, measurement of the viscosity of the slurry can be used to estimate the amount of fines in the oil sands slurry and therefore used to control, for example, the amount of bridging added to the slurry. This measurement can be obtained in a simple viscometer or in a rheometer. Other related tests can also be used, such as a flow rate test.

[0059] In another embodiment, measurement of the rheology of the slurry can be
10 used to determine the progression of the agglomeration process. For example, after the bridging liquid is added to the slurry and agitated, a rapid increase in the viscosity of the slurry may indicate excessive agglomerate growth that has led to the trapping of a significant amount of bitumen extract within the agglomerates. Conditions that lead to such behavior should be limited or avoided since they can lead to poor bitumen recovery. The control
15 system described herein can be used to change process parameters, such as the amount of bridging liquid addition, when the viscosity of the slurry is measured to rapidly increase. In another example, the viscosity or rheometer measurement can be used to track the growth of agglomerates. In cases when the formed agglomerates are compact, the growth of agglomerates may be accompanied by a gradual reduction in slurry viscosity or dynamic
20 shear strength. Thus, the change in slurry viscosity may correlate well with agglomerate growth.

[0060] The viscosity of the oil sand slurry may be measured with any suitable instrument that is well known in the art. For example, an automatic on-line viscometer, which takes a slip stream from the slurry and measures the viscosity, can be used. An in-
25 line viscometer, such as a vibrating-type viscometer, can be used to provide instant viscosity measurements within the process slurry. In another example, the torque is measured in the agitation process, and rheological measurements could be determined in-situ. That is, if a mixing vessel is used for the agglomerator, the torque applied to the vessel can be measured as an indicator of rheological properties such as viscosity.

30 **[0061]** Various other properties of the bituminous feed, or the outputs could be alternatively or additionally be measured.

[0062] In another embodiment, the following steps may be performed:

1. Drill ore cores in advance of mining trucks to determine the quality of the ore.

2. As shovels proceed through a seam, obtain further data to characterize the ore. Send the ore to the extraction process, characterized as low, medium, or high fines content, or along another rating system.

3. Combine the oil sands with an extraction liquor and a bridging liquid in an agglomerator. The extraction liquor comprises a solvent used to dissolve bitumen. The bridging liquid is used to assist agglomeration. The bridging liquid may be water or a sludge from a water-based extraction process. Suitable sludge streams include, but are not limited to, water-based extraction streams such as middling from primary separation, secondary and tertiary separation tailings, froth treatment tailings, mature fine tailings from tailings ponds, or a new stream resulting from passing any of these streams through a thickener, hydrocyclone, or other processes. For example, middlings passed through a cyclone might generate an overflow stream and an underflow stream. Either stream could be used in this process as bridging liquid. The amount of bridging liquid that is added will affect the extent of agglomeration. Agitation is also used to assist agglomeration.

4. Adjust one or more process parameters based on one or more output properties. One process parameter is the amount of bridging liquid that is added to the slurry. Another process parameter is the solid content of the bridging liquid added to the slurry. Another process parameter is the methods and locations of the bridging liquid addition in the process. Another process parameter is the solid content of the slurry. Another process parameter is the intensity of agitation of the slurry. Another process parameter is the shear environment of the agglomerator. Another process parameter is the residence time of the extraction process. Yet another option process parameter is the residence time of the agglomeration process. Potential output properties include particle size distribution of the produced agglomerates, filtration rate of the slurry, solids content of the low solids bitumen extract, bitumen content of low solids bitumen extract, and the viscosity of the slurry. The adjustments to the process parameters may be made based on the real time measurements of physical properties of the output(s) of the agglomeration process, which result in a feedback. In one embodiment, the feedback loop is a negative feedback, since the desired outputs of the agglomeration process may be set to one or more given target ranges and the input parameters may be adjusted to maintain the output parameters in the target range(s) regardless of type of ore feed and process upsets. The expression "target range" as used herein may include a range such as between X and Y, but also may include a range such as at least Z, or a range such as less than W.

[0063] In one embodiment, the characterization of fines content comprises a

methylene blue test. In another embodiment, the characterization of fines comprises a particle size distribution analysis. In another embodiment, the characterization of fines comprises viscosity/rheology tests of oil sands slurry. In another embodiment, the ore (or bituminous feed) is characterized by bitumen content rather than, or in addition to, fines content. In another embodiment, the ore (or bituminous feed) is characterized by spectroscopy, photoluminescence, fluorescence, or other photoactive technology. In another embodiment, the ore (or bituminous feed) is characterized by water chemistry and/or quantity. In yet another embodiment, the output solids are characterized by particle size distribution using sieves, laser diffraction, optical analysis, or other size quantification technique. In another embodiment, the hydrocarbon content of the output stream is measured by a bomb calorimeter, gas chromatography, photo activity such as phosphorescence or other photon technique, particle sniffer, or other technology. In another embodiment, the moisture content is measured by any type of technique suitable to measure water content, including but not limited to a bomb calorimeter, Karl Fischer Titration, Deen Stark analysis, electrical conductivity, relative humidity, or any other technique. In one embodiment, the analysis is performed in conjunction with batch analysis at intervals. In another embodiment, a slip stream is sampled for analysis. In another embodiment, on-line analysis provides continuous information.

[0064] In another embodiment, the bridging liquid is adjusted based on a measured property. The following steps may be performed, with reference to Figure 4:

1. Adding the slurry (**402**) comprised of bituminous feed and extraction liquor to an agglomerator (**404**).
2. Providing two different streams of bridging liquid (**406** and **408**) to the agglomerator (**404**) to form an agglomerated slurry (**410**).
3. Based on information on the quality of the oil sand ore (or bituminous feed) or the quality of one or more output streams (i.e. the low solids bitumen extract or the agglomerates) or both, adjusting (using a control point (**412**)) one or both of the flow rates of bridging liquid.

[0065] In one embodiment, the first bridging liquid comprises water and the second bridging liquid comprises sludge produced from the aqueous extraction of bitumen from oil sands.

[0066] In another embodiment, as shown in Figure 5, first and second bridging liquids are mixed before they are introduced into the agglomerator. The slurry comprised of

bituminous feed and extraction liquor (together **502**) is added to an agglomerator (**504**). The first bridging liquid (**506**) and second bridging liquid (**508**) are mixed to form a mixed bridging liquid (**514**) and added to the agglomerator (**504**) to form an agglomerated slurry (**510**). Based on information on the quality of the oil sand ore (or bituminous feed) or the quality of one or more output streams (i.e. the low solids bitumen extract or the agglomerates) or both, one or both of the flow rates of bridging liquids (**506** and **508**) are adjusted (using a control point (**512**)). In yet another embodiment, the first and second bridging liquids are mixed in the agglomerator.

[0067] In another embodiment, as shown in Figure 6, the properties of the agglomeration process are adjusted through the recycling of agglomerator output upstream of the agglomeration process. For example, the agglomerated slurry could be recycled through the process to affect the residence time of the agglomeration process. The agglomerated solids could also be recycled through the process to increase the solids content of the feed slurry. Additionally, the agglomerated solids could be recycled through the process to provide seed particles within the bridging liquid for the agglomeration process. First, properties of the bituminous feed and extraction liquor (**602**) are measured (A). In another embodiment, the bituminous feed may be measured prior to contact with the extraction liquor. The bridging liquid (**604**) is added to the agglomerator (**606**) to produce outputs (**608**) (i.e. the agglomerates and the low solids bitumen extract) of the agglomeration process. One or more properties of the outputs are measured (B). The measurements may be performed continuously. The measurements (A and B) are used in a control system (**610**) to adjust a parameter of the process, for instance the amount and/or composition of an input. For instance, a portion of the agglomerated solids (**611**) could be recycled back into the process to adjust effective residence time and/or increase solids content.

[0068] In another embodiment, the at least one property further comprises at least one property of the slurry prior to agglomeration.

[0069] Agitation. Agglomeration is assisted by some form of agitation. The form of agitation may be mixing, shaking, rolling, or another known suitable method. The agitation of the feed need only be severe enough and of sufficient duration to intimately contact the emulsion with the solids in the feed. Exemplary rolling type vessels include rod mills and tumblers. Exemplary mixing type vessels include mixing tanks, blenders, and attrition scrubbers. In the case of mixing type vessels, a sufficient amount of agitation is needed to keep the formed agglomerates in suspension. In rolling type vessels, the solids content of

the feed is, in one embodiment, greater than 40 wt.% so that compaction forces assist agglomerate formation. The agitation of the slurry has an impact on the growth of the agglomerates. In the case of mixing type vessels, the mixing power can be increased in order to limit the growth of agglomerates by attrition of said agglomerates. In the case of
5 rolling type vessels the fill volume and rotation rate of the vessel can be adjusted in order to increase the compaction forces used in the comminution of agglomerates. These agitation parameters can be adjusted in the control system described herein.

[0070] Extraction Liquor. The extraction liquor comprises a solvent used to extract bitumen from the bituminous feed. The term "solvent" as used herein should be understood
10 to mean either a single solvent, or a combination of solvents.

[0071] In one embodiment, the extraction liquor comprises a hydrocarbon solvent capable of dissolving the bitumen. The extraction liquor may be a solution of a hydrocarbon solvent(s) and bitumen, where the bitumen content of the extraction liquor may range between 10 to 50 wt%. It may be desirable to have dissolved bitumen within the extraction
15 liquor in order to increase the volume of the extraction liquor without an increase in the required inventory of hydrocarbon solvent(s). In cases where non-aromatic hydrocarbon solvents are used, the dissolved bitumen within the extraction liquor also increases the solubility of the extraction liquor towards dissolving additional bitumen.

[0072] The extraction liquor may be mixed with the bituminous feed to form a slurry
20 where most or all of the bitumen from the oil sands is dissolved into the extraction liquor. In one embodiment, the solids content of the slurry is in the range of 10 wt% to 75 wt%, or 50 to 65 wt%. A slurry with a higher solids content may be more suitable for agglomeration in a rolling type vessel, where the compressive forces aid in the formation of compact agglomerates. For turbulent flow type vessels, such as an attrition scrubber, a slurry with a
25 lower solids content may be more suitable.

[0073] The solvent used in the process may include low boiling point solvents such as low boiling point cycloalkanes, or a mixture of such cycloalkanes, which substantially dissolve asphaltenes. The solvent may comprise a paraffinic solvent in which the solvent to bitumen ratio is maintained at a level to avoid or limit precipitation of asphaltenes.

[0074] While it is not necessary to use a low boiling point solvent, when it is used, there is the extra advantage that solvent recovery through an evaporative process proceeds at lower temperatures, and requires a lower energy consumption. When a low boiling point solvent is selected, it may be one having a boiling point of less than 100 °C.
30

[0075] The solvent selected according to certain embodiments may comprise an organic solvent or a mixture of organic solvents. For example, the solvent may comprise a paraffinic solvent, an open chain aliphatic hydrocarbon, a cyclic aliphatic hydrocarbon, or a mixture thereof. Should a paraffinic solvent be utilized, it may comprise an alkane, a natural
5 gas condensate, a distillate from a fractionation unit (or diluent cut), or a combination of these containing more than 40% small chain paraffins of 5 to 10 carbon atoms. These embodiments would be considered primarily a small chain (or short chain) paraffin mixture. Should an alkane be selected as the solvent, the alkane may comprise a normal alkane, an iso-alkane, or a combination thereof. The alkane may specifically comprise heptane, iso-
10 heptane, hexane, iso-hexane, pentane, iso-pentane, or a combination thereof. Should a cyclic aliphatic hydrocarbon be selected as the solvent, it may comprise a cycloalkane of 4 to 9 carbon atoms. A mixture of C₄-C₉ cyclic and/or open chain aliphatic solvents would be appropriate.

[0076] Exemplary cycloalkanes include cyclohexane, cyclopentane, or a mixture
15 thereof.

[0077] If the solvent is selected as the distillate from a fractionation unit, it may for example be one having a final boiling point of less than 180 °C. An exemplary upper limit of the final boiling point of the distillate may be less than 100 °C.

[0078] A mixture of C₄-C₁₀ cyclic and/or open chain aliphatic solvents would also be
20 appropriate. For example, it can be a mixture of C₄-C₉ cyclic aliphatic hydrocarbons and paraffinic solvents where the percentage of the cyclic aliphatic hydrocarbons in the mixture is greater than 50%.

[0079] Extraction liquor may be recycled from a downstream step. For instance, as described below, solvent recovered in a solvent recovery unit, may be used to wash
25 agglomerates, and the resulting stream may be used as extraction liquor. As a result, the extraction liquor may comprise residual bitumen and residual solid fines. The residual bitumen increases the volume of the extraction liquor and it may increase the solubility of the extraction liquor for additional bitumen dissolution.

[0080] The solvent may also include additives. These additives may or may not be
30 considered a solvent per se. Possible additives may be components such as de-emulsifying agents or solids aggregating agents. Having an agglomerating agent additive present in the bridging liquid and dispersed in the first solvent may be helpful in the subsequent agglomeration step. Exemplary agglomerating agent additives include cements, fly ash,

gypsum, lime, brine, water softening wastes (e.g. magnesium oxide and calcium carbonate), solids conditioning and anti-erosion aids such as polyvinyl acetate emulsion, commercial fertilizer, humic substances (e.g. fulvic acid), polyacrylamide based flocculants and others. Additives may also be added prior to gravity separation with the second solvent to enhance
5 removal of suspended solids and prevent emulsification of the two solvents. Exemplary additives include methanoic acid, ethylcellulose and polyoxyalkylate block polymers.

[0081] ***Bridging Liquid.*** A bridging liquid is a liquid with affinity for the solids particles in the bituminous feed, and which is immiscible in the solvent. Exemplary aqueous liquids may be recycled water from other aspects or steps of oil sands processing. The aqueous
10 liquid need not be pure water, and may indeed be water containing one or more salt, a waste product from conventional aqueous oil sand extraction processes which may include additives, aqueous solutions with a range of pH, or any other acceptable aqueous solution capable of adhering to solid particles within an agglomerator in such a way that permits fines to adhere to each other. An exemplary bridging liquid is water.

15 **[0082]** The total amount of bridging liquid added to the slurry may be controlled in order to optimize bitumen recovery and the rate of solid-liquid separation. The value will depend on the measured properties described herein. By way of examples, the total amount of bridging liquid added to the slurry may be such that a ratio of bridging liquid plus connate water from the bituminous feed to solids within the agglomerated slurry is in the range of 0.02
20 to 0.25, or in the range of 0.05 to 0.11. In one embodiment, the bridging liquid to solids ratio may be obtained by feedback control.

[0083] In one embodiment, the bridging liquid may contain fine particles (sized less than 44 μm) suspended therein. These fine particles may serve as seed particles for the agglomeration process. In one embodiment, the bridging liquid has a solids content of less
25 than 40 wt.%. The bridging liquid and fines particles slurry is also referred to herein as sludge from a water-based extraction process. Suitable sludge streams include, but are not limited to, water-based extraction streams such as middling from primary separation, secondary and tertiary separation tailings, froth treatment tailings, mature fine tailings from tailings ponds, or a new stream resulting from passing any of these streams through a
30 thickener, hydrocyclone, or other processes. For example, middlings passed through a cyclone might generate an overflow stream and an underflow stream. Either stream could be used in this process as bridging liquid. Sludge may also be produced within the solvent extraction with solids agglomeration process by mixing bridging liquid with agglomerated tailings. In this way, a portion of the agglomerated solids are recycled through the process.

The use of bridging liquid with a significant solid content, such as that which is described above, may allow for greater control of the agglomeration process. Previous work has shown that when sludge is used as the bridging liquid, the addition of the same amount of sludge per unit weight of oil sands feed resulted in the production of agglomerates of the same drainage properties regardless of oil sands quality.

[0084] The bridging liquid may be added after the production of the oil sands slurry or before the production of the oil sands slurry. In the former scenario, the bitumen is first extracted from the bituminous feed prior to agglomeration in order to prevent (or limit) the agglomeration process from hampering the dissolution of bitumen into the extraction liquor, which may increase bitumen recovery. In the latter scenario, the bridging liquid may be directly mixed with the bituminous feed before or at the same time as the extraction liquor in order to minimize the dispersion of fines, which may reduce the solids content of the bitumen extract after the agglomeration process. The control system described herein can be used to control where in the solvent extraction with solids agglomeration process the bridging liquid is added based on the output of the process. The bridging liquid may comprise less than 40 wt% solids fines. The agglomerated slurry may have a solids content of 20 to 70 wt%.

[0085] Ratio of Solvent to Bitumen for Agglomeration. The process may be adjusted to render the ratio of the solvent to bitumen in the agglomerator at a level that avoids precipitation of asphaltenes during agglomeration. Some amount of asphaltene precipitation is unavoidable, but by adjusting the amount of solvent flowing into the system, with respect to the expected amount of bitumen in the bituminous feed, when taken together with the amount of bitumen that may be entrained in the extraction liquor used, can permit the control of a ratio of solvent to bitumen in the agglomerator. When the solvent is assessed for an optimal ratio of solvent to bitumen during agglomeration, the precipitation of asphaltenes can be minimized or avoided beyond an unavoidable amount. Another advantage of selecting an optimal solvent to bitumen ratio is that when the ratio of solvent to bitumen is too high, costs of the process may be increased due to increased solvent requirements.

[0086] An exemplary ratio of solvent to bitumen to be selected as a target ratio during agglomeration is less than 2:1. A ratio of 1.5:1 or less, and a ratio of 1:1 or less, for example, a ratio of 0.75:1, would also be considered acceptable target ratios for agglomeration. For clarity, ratios may be expressed herein using a colon between two values, such as "2:1", or may equally be expressed as a single number, such as "2", which

carries the assumption that the denominator of the ratio is 1 and is expressed on a weight to weight basis.

5 [0087] Measurement of the solvent and bitumen content of the extraction liquor and/or bitumen extract could occur directly or by proxy. Direct measurement of solvent and bitumen content could involve evaporating off the solvent and measuring the mass of both liquids, or use of a gas chromatograph, mass balance, spectrometer, or titration. Indirect measurement of solvent and bitumen content could include measuring density, the index of refraction, opacity, or other properties.

10 [0088] **Slurry System.** The slurry system may optionally be a mix box, a pump, or a combination of these. By slurrying the extraction liquor together with the bituminous feed, and optionally with additional additives, the bitumen entrained within the feed is given an opportunity to become extracted into the solvent phase prior to agglomeration within the agglomerator.

15 [0089] The resulting slurry from the slurry system may have a solid content in the range of 20 to 65 wt%. In another embodiment, the slurry may have a solid content in the range of 20 to 50 wt%. In another embodiment, the slurry may have a solid content in the range of 40 to 65 wt%. In the case of mixing type vessels, a lower solid content may be preferred since that will assist in the proper mixing of the bridging liquid and reduce the mixing energy needed to keep the slurry well mixed. In the case of rolling type vessels, a
20 higher solid content may be preferred since that will increase the compaction forces used in the comminution of agglomerates. Additionally, the increased compaction forces may reduce the amount of hydrocarbons that remain in the agglomerates and produce stronger agglomerates.

25 [0090] The preferred temperature of the slurry is in the range of 20-60 °C. An elevated slurry temperature is desired in order to increase the bitumen dissolution rate and reduce the viscosity of the slurry to promote more effective sand digestion and agglomerate formation. Temperatures above 60 °C are generally avoided due to the complications resulting from high vapor pressures.

30 [0091] **Residence Time.** The residence time of the extraction and agglomeration processes has a strong impact on the bitumen extract and agglomerated solids. Batch experiments within a mixing vessel were conducted to test the affects of residence time. Figure 8 (as described further below) shows that the bitumen recovery and the initial liquid filtration rate increases as the extraction time increases for batch experiments conducted

with the agglomeration time kept constant at 2 minutes. Thus, increasing the residence time of the extraction process may result in an increase in both the bitumen recovery and the rate of solid-liquid separation. In contrast, as Figure 9 (as described further below) shows, the bitumen recovery reaches a maximum and then decreases as the agglomeration time increases for batch experiments conducted with the extraction time kept constant at 5 minutes. The decrease in recovery beyond the maximum recovery is most likely due to excessive agglomerate growth that leads to entrapment of the bitumen extract within the agglomerates. However, this growth of agglomerates does result in an increase in the initial filtration rate as the agglomeration time increases.

10 **[0092]** The results plotted in Figure 8 and Figure 9 demonstrate the impact that residence time of the extraction and agglomeration processes have on the bitumen extract and agglomerated solids.

[0093] As shown in Figure 10, the recycle loops, (1020) and (1022) can be used in the control system described herein to adjust the effective residence time within the slurry system (1005) and agglomerator (1006). First, properties of the bituminous feed and extraction liquor (1002) are measured (A). In another embodiment, the bituminous feed may be measured prior to contact with the extraction liquor. The bridging liquid (1004) is added to the slurry system (1005) and the slurry is passed to the agglomerator (1006) to produce outputs (1008) (i.e. the agglomerates and the low solids bitumen extract) of the agglomeration process. One or more properties of the outputs (1008) are measured (B). The measurements may be performed continuously. The measurements (A and B) are used in a control system (1010) to adjust a parameter of the process, for instance the amount and/or composition of an input. For instance, a portion of the agglomerated solids (1022) or a portion of the slurry prior to agglomeration (1020) could be recycled back into the process to adjust effective residence time and/or increase solids content.

[0094] The results plotted in Figure 8 and Figure 9 also suggest that it is preferable for the residence time of the extraction process be greater or much greater than the residence time of the agglomeration process. The extraction process may occur in the slurry system and the agglomeration process may occur in the agglomerator. The residence time of the extraction process may be greater than 5 minutes, or may be greater than 10 minutes, or may be greater than 15 minutes, or may be greater than 30 minutes. Depending on the desired level of agglomeration, the residence time of the agglomeration process may be in the range of 15 seconds to 10 minutes. In order to maximize bitumen recovery, the residence time of the agglomeration process may be in the range of 1 to 5 minutes.

[0095] Solid-Liquid Separator. As described above, the agglomerated slurry may be separated into a low solids bitumen extract and agglomerates in a solid-liquid separator. The solid-liquid separator may comprise any type of unit capable of separating solids from liquids, so as to remove agglomerates. Exemplary types of units include a gravity separator,
5 a clarifier, a cyclone, a screen, a belt filter or a combination thereof.

[0096] The system may contain a solid-liquid separator but may alternatively contain more than one. When more than one solid-liquid separation step is employed at this stage of the process, it may be said that both steps are conducted within one solid-liquid separator, or if such steps are dissimilar, or not proximal to each other, it may be said that a primary
10 solid-liquid separator is employed together with a secondary solid-liquid separator. When a primary and secondary unit are both employed, generally, the primary unit separates agglomerates, while the secondary unit involves washing agglomerates.

[0097] Non-limiting methods of solid-liquid separation of an agglomerated slurry are described in Canadian Patent Application Serial No. 2,724,806 (Adeyinka et al.), filed
15 December 10, 2010.

[0098] Secondary Stage of Solid-Liquid Separation to Wash Agglomerates. As a component of the solid-liquid separator, a secondary stage of separation may be introduced for countercurrently washing the agglomerates separated from the agglomerated slurry. The initial separation of agglomerates may be said to occur in a primary solid-liquid
20 separator, while the secondary stage may occur within the primary unit, or may be conducted completely separately in a secondary solid-liquid separator. By "countercurrently washing", it is meant that a progressively cleaner solvent is used to wash bitumen from the agglomerates. Solvent involved in the final wash of agglomerates may be re-used for one or more upstream washes of agglomerates, so that the more bitumen entrained on the
25 agglomerates, the less clean will be the solvent used to wash agglomerates at that stage. The result being that the cleanest wash of agglomerates is conducted using the cleanest solvent.

[0099] A secondary solid-liquid separator for countercurrently washing agglomerates may be included in the system or may be included as a component of a system described
30 herein. The secondary solid-liquid separator may be separate or incorporated within the primary solid-liquid separator. The secondary solid-liquid separator may optionally be a gravity separator, a cyclone, a screen or belt filter. Further, a secondary solvent recovery unit for recovering solvent arising from the solid-liquid separator can be included. The secondary solvent recovery unit may be a conventional fractionation tower or a distillation

unit.

[0100] When conducted in the process, the secondary stage for countercurrently washing the agglomerates may comprise a gravity separator, a cyclone, a screen, a belt filter, or a combination thereof.

- 5 **[0101]** The solvent used for washing the agglomerates may be solvent recovered from the low solids bitumen extract, as described with reference to Figures 2 to 4. A second solvent may alternatively or additionally be used as described in Canadian Patent Application Serial No. 2,724,806 (Adeyinka et al.) for additional bitumen extraction downstream of the agglomerator.

- 10 **[0102] **Recycle and Recovery of Solvent.**** The process may involve removal and recovery of solvent used in the process.

[0103] In this way, solvent is used and re-used, even when a good deal of bitumen is entrained therein. Because an exemplary solvent:bitumen ratio in the agglomerator may be 2:1 or lower, it is acceptable to use recycled solvent containing bitumen to achieve this ratio.

- 15 The amount of make-up solvent required for the process may depend solely on solvent losses, as there is no requirement to store and/or not re-use solvent that has been used in a previous extraction step. When solvent is said to be "removed", or "recovered", this does not require removal or recovery of all solvent, as it is understood that some solvent will be retained with the bitumen even when the majority of the solvent is removed.

- 20 **[0104]** The system may contain a single solvent recovery unit for recovering the solvent(s) arising from the gravity separator. The system may alternatively contain more than one solvent recovery unit.

- [0105]** Solvent may be recovered by conventional means. For example, typical solvent recovery units may comprise a fractionation tower or a distillation unit. The solvent recovered in this fashion will not contain bitumen entrained therein. This clean solvent is preferably used in the last wash stage of the agglomerate washing process in order that the cleanest wash of the agglomerates is conducted using the cleanest solvent.
- 25

- [0106]** The solvent recovered in the process may comprise entrained bitumen therein, and can thus be re-used as the extraction liquor for combining with the bituminous feed. Other optional steps of the process may incorporate the solvent having bitumen entrained therein, for example in countercurrent washing of agglomerates, or for adjusting the solvent and bitumen content prior to agglomeration to achieve the selected ratio within
- 30

the agglomerator that avoids precipitation of asphaltenes.

[0107] Dilution of Agglomerator Discharge to Improve Product Quality. Solvent may be added to the agglomerated slurry for dilution of the slurry before discharge into the primary solid-liquid separator, which may be for example a deep cone settler. This dilution
5 can be carried out in a staged manner to pre-condition the primary solid-liquid separator feed to promote higher solids settling rates and lower solids content in the solid-liquid separator's overflow. The solvent with which the slurry is diluted may be derived from recycled liquids from the liquid-solid separation stage or from other sources within the process.

[0108] When dilution of agglomerator discharge is employed in this embodiment, the
10 solvent to bitumen ratio of the feed into the agglomerator is set to obtain from about 10 to about 90 wt% bitumen in the discharge, and a workable viscosity at a given temperature. In certain cases, these viscosities may not be optimal for the solid-liquid separation (or settling) step. In such an instance, a dilution solvent of equal or lower viscosity may be added to enhance the separation of the agglomerated solids in the clarifier, while improving the quality
15 of the clarifier overflow by reducing viscosity to permit more solids to settle. Thus, dilution of agglomerator discharge may involve adding the solvent, or a separate dilution solvent, which may, for example, comprise an alkane.

[0109] In the preceding description, for purposes of explanation, numerous details
20 are set forth in order to provide a thorough understanding of the embodiments. However, it will be apparent to one skilled in the art that these specific details are not required.

[0110] Control Systems. Embodiments of the disclosure can be represented as a computer program product stored in a machine-readable medium (also referred to as a computer-readable medium, a processor-readable medium, or a computer usable medium having a computer-readable program code embodied therein). The machine-readable
25 medium can be any suitable tangible, non-transitory medium, including magnetic, optical, or electrical storage medium including a diskette, compact disk read only memory (CD-ROM), memory device (volatile or non-volatile), or similar storage mechanism. The machine-readable medium can contain various sets of instructions, code sequences, configuration information, or other data, which, when executed, cause a processor to perform steps in a
30 method according to an embodiment of the disclosure. Those of ordinary skill in the art will appreciate that other instructions and operations necessary to implement the described implementations can also be stored on the machine-readable medium. The instructions stored on the machine-readable medium can be executed by a processor or other suitable processing device, and can interface with circuitry to perform the described tasks.

[0111] The above-described embodiments are intended to be examples only. Alterations, modifications and variations can be effected to the particular embodiments by those of skill in the art without departing from the scope, which is defined solely by the claims appended hereto.

5 [0112] **Batch Experiments.** Experiments were conducted to test the effects of varying residence time on the extraction and agglomeration processes. The initial liquid drainage rate of the formed agglomerates and bitumen recovery from the oil sands were used as the experimental measurements to determine the effectiveness of the solvent extraction with solids agglomeration process. The agglomerates were also visually
10 inspected for their size and uniformity.

[0113] Medium grade Athabasca oil sand was used in these experiments. The oil sands had a bitumen content of 9.36 wt% and a water content of 4.66 wt%. The percentage of fines ($< 44 \mu\text{m}$) that make up the solids was approximately 25 wt%. The oil sands were kept at -20°C until they were ready for use. A solution of cyclohexane and bitumen was
15 used as the extraction liquor. The percentage of bitumen in the extraction liquor was 24 wt%. Distilled water was used as the bridging liquid. For each experiment a total of 350g of oil sands, 235.07g of extraction liquor, and a total of 16.8g of water were used. This composition translated to a solids content of 50 wt% and a water to solids ratio of 0.11 for the agglomerated slurry.

20 [0114] A Parr reactor (series 5100) (Parr Instrument Company, Moline, IL, USA) was used as the extractor and agglomerator. The reactor vessel was made of glass that permits direct observation of the mixing process. A turbine type impeller powered by an explosion proof motor of 0.25 hp was used. The mixing and agglomeration speed of the impeller were set to 1500 rpm. This rotation speed allowed the slurry to remain fluidized at all conditions of
25 the experiments. The agglomeration experiments were conducted at room temperature (22°C).

[0115] The agglomerated solids produced in these experiments were treated in a Soxhlet extractor combined with Dean-Stark azeotropic distillation, to determine the material contents of the agglomerated slurry. Toluene was used as the extraction solvent. The oil
30 sand solids were dried overnight in an oven (100°C) and then weighed to determine the solids content of the agglomerated slurry. The water content was determined by measuring the volume of the collected water within the side arm of the Dean-Stark apparatus. The bitumen content of the agglomerated slurry was determined by evaporating the toluene and residual cyclohexane from an aliquot of the hydrocarbon extract from the Soxhlet extractor.

[0116] The initial liquid drainage rate was calculated by measuring the time needed to drain 50 mL of bitumen extract above the bed of agglomerated solids.

[0117] **The effects of extraction residence time on the solvent extraction with solids agglomeration process.** 350g of oil sands and 235.07g of extraction liquor were placed into the Parr reactor vessel. The solids and solvent were mixed at 1500 rpm for a given extraction residence time to homogenize the mixture and to extract the bitumen that was in the oil sands. The extraction times tested were 0.5, 1, 2, 5, 15, and 30 minutes. After the extraction time elapsed, 16.8g of water was quickly pored into the vessel through a sample port. The mixture was then mixed at 1500 rpm for an additional 2 minutes to agglomerate the solids.

[0118] After the agglomeration process, the impeller was turned off and the agglomerates were allowed to settle for over 1 minute. The supernatant (bitumen extract) was poured into a separate container and the wet solids were transferred to a Buchner funnel. The solids rested on a filter paper with a nominal pore size of 170 μm . The filter's effective area was approximately 8 cm^2 . The solids bed height was 10.8 cm. A portion of the collected supernatant was poured on top of the solids until a liquid height of 1.9 cm formed above the solids surface. A light vacuum was then applied to the Buchner funnel and the initial drainage rate of the liquid was recorded.

[0119] The remaining supernatant was poured onto the solid bed and allowed to filter through. 211 mL of pure cyclohexane was then filtered through the solid bed in order to wash the agglomerates. The solid bed was then allowed to drain of liquid under a light vacuum for about 30 seconds. The bitumen content of the washed solids was then measured to determine the bitumen recovery of the solvent extraction process.

[0120] Figure 8 plots the bitumen recovery and the initial liquid filtration rate as a function of the extraction residence time. The figure shows that the bitumen recovery and the initial liquid filtration rate increases as the extraction time increases for batch experiments conducted with the agglomeration time kept constant at 2 minutes.

[0121] **The effects of agglomeration residence time on the solvent extraction with solids agglomeration process.** 350g of oil sands and 235.07g of extraction liquor were placed into the Parr reactor vessel. The solids and solvent were mixed at 1500 rpm for 5 minutes to fully homogenize the mixture and to fully extract the bitumen that was in the oil sands. After 5 minutes of mixing, 16.8g of water was quickly pored into the vessel through a sample port. The mixture was then mixed at 1500 rpm for a given agglomeration residence

time to agglomerate the solids. The agglomeration times tested were 0.5, 1, 2, 5, 15, and 30 minutes.

[0122] After the agglomeration process, the impeller was turned off and the agglomerates were allowed to settle for over 1 minute. The supernatant (bitumen extract) was pored into a separate container and the wet solids were transferred to a Buchner funnel. The solids rested on a filter paper with a nominal pore size of 170 μm . The filter's effective area was approximately 8 cm^2 . The solids bed height was 10.8 cm. A portion of the collected supernatant was poured on top of the solids until a liquid height of 1.9 cm formed above the solids surface. A light vacuum was then applied to the Buchner funnel and the initial drainage rate of the liquid was recorded.

[0123] The remaining supernatant was poured onto the solid bed and allowed to filter through. 211 mL of pure cyclohexane was then filtered through the solid bed in order to wash the agglomerates. The solid bed was then allowed to drain of liquid under a light vacuum for about 30 seconds. The bitumen content of the washed solids was then measured to determine the bitumen recovery of the solvent extraction process.

[0124] Figure 9 plots the bitumen recovery and the initial liquid filtration rate as a function of the agglomeration residence time. The figure shows that the bitumen recovery reaches a maximum and then decreases as the agglomeration time increases for batch experiments conducted with the extraction time kept constant at 5 minutes. The decrease in recovery beyond the maximum recovery is most likely due to excessive agglomerate growth that lead to entrapment of the bitumen extract within the agglomerates. However, this growth of agglomerates does result in a continuous increase in the initial filtration rate as the agglomeration time increases.

CLAIMS

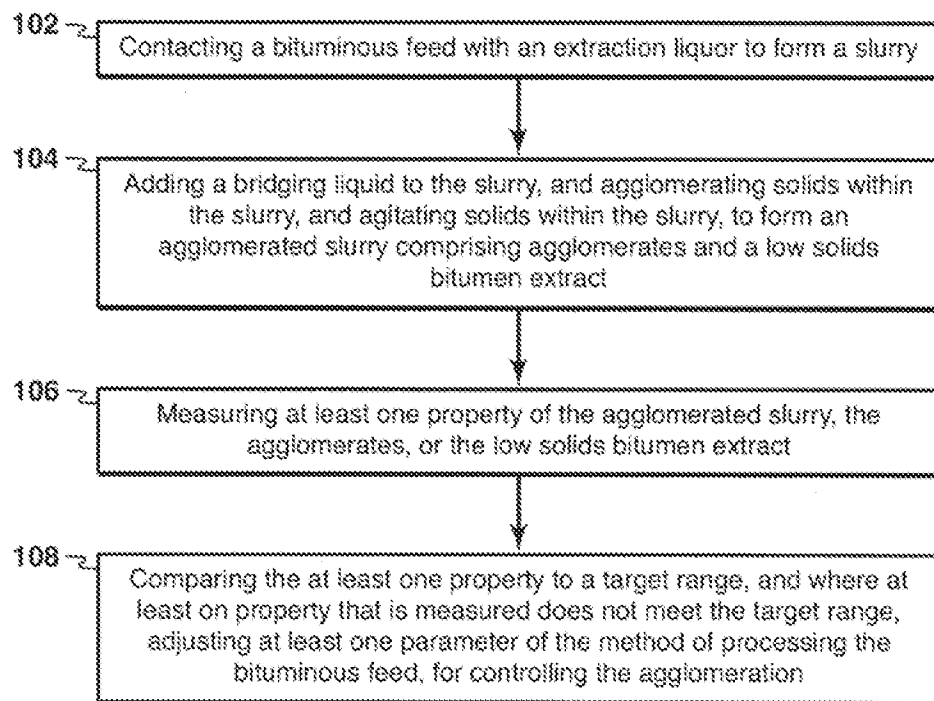
What is claimed is:

1. A method of processing a bituminous feed, the method comprising:
 - a) contacting the bituminous feed with an extraction liquor to form a slurry,
5 wherein the extraction liquor comprises a solvent;
 - b) adding a bridging liquid to the slurry, and agitating solids within the slurry, to form an agglomerated slurry comprising agglomerates and a low solids bitumen extract;
 - c) measuring at least one property of the agglomerated slurry, the agglomerates, or the low solids bitumen extract; and
10 d) comparing the at least one property to a target range, and where the at least one property that is measured does not fall within the target range, adjusting at least one parameter of the method of processing the bituminous feed, for controlling the agglomeration.
2. The method of claim 1, wherein the at least one property further comprises at least
15 one property of the slurry prior to agglomeration or at least one property of the bituminous feed.
3. The method of claim 1, wherein the at least one property comprises a fines content of the low solids bitumen extract, clay chemistry, a rheological property of the agglomerated slurry, a particle size distribution of the agglomerates, a filtration rate of the agglomerated
20 slurry, or bitumen content of the low solids bitumen extract.
4. The method of claim 1, wherein the at least one parameter comprises an amount of added bridging liquid relative to an amount of added bituminous feed, a composition of the bridging liquid, or a position at which at least a portion of the bridging liquid is added to the slurry.
- 25 5. The method of claim 1, wherein the at least one parameter comprises an extraction residence time.
6. The method of claim 5, wherein the extraction residence time is greater than 5 minutes.

7. The method of claim 1, wherein the at least one parameter comprises an agglomeration residence time.
8. The method of claim 7, wherein the agglomeration residence time is 1 to 5 minutes.
9. The method of claim 1, wherein the at least one parameter comprises an amount of
5 extraction liquor added relative to an amount of added bituminous feed, a level of agitation provided to the slurry, or a shear environment of the agglomeration.
10. The method of claim 2, wherein the at least one property of the bituminous feed comprises water content.
11. The method of claim 2, wherein the at least one property of the bituminous feed
10 comprises insoluble inorganics content.
12. The method of claim 1, wherein the adding the bridging liquid to the slurry comprises adding a combination of at least two different bridging liquid streams.
13. The method of claim 12, wherein the at least one parameter comprises relative
15 amounts of the at least two different bridging liquid streams, recycling at least a portion of the agglomerated slurry into step a), recycling at least a portion of the agglomerated slurry between steps a) and b), or recycling at least a portion of the agglomerates into step b).
14. The method of claim 1, wherein the bridging liquid is added to the slurry in a concentration of between 1 and 20 wt% of the slurry.
15. The method of claim 1, the bridging liquid is added to the slurry in a concentration of
20 between 1 and 10 wt% of the slurry.
16. The method of claim 1, further comprising separating the agglomerates from the low solids bitumen extract.
17. The method of claim 16, further comprising recovering the solvent from the low solids bitumen extract to form a bitumen product.
- 25 18. The method of claim 1, wherein the extraction liquor comprises the solvent of step a) and bitumen in an amount of 10 to 50 wt%.
19. The method of claim 1, wherein the bridging liquid is water.
20. The method of claim 1, wherein the bridging liquid is an aqueous solution.

21. The method of claim 1, wherein at least 80 wt. % of the agglomerates of step c) are between 0.1 and 1 mm.
22. The method of claim 1, wherein the agglomerated slurry has a solids content of 20 to 70 wt%.
- 5 23. The method of claim 1, wherein the solvent comprises an organic solvent or a mixture of organic solvents.
24. The method of claim 1, wherein the solvent comprises at least 50 wt.% cyclohexane.
25. The method of claim 1, wherein the agglomeration is effected in one or more vessels.
26. The method of claim 1, wherein step b) comprises agitating by mixing, shaking, or
10 rolling.
27. The method of claim 1, wherein a ratio of the solvent to bitumen in the agglomerated slurry is less than 2:1.
28. The method of claim 1, wherein the bituminous feed is derived from oil sands.

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**FIG. 1**

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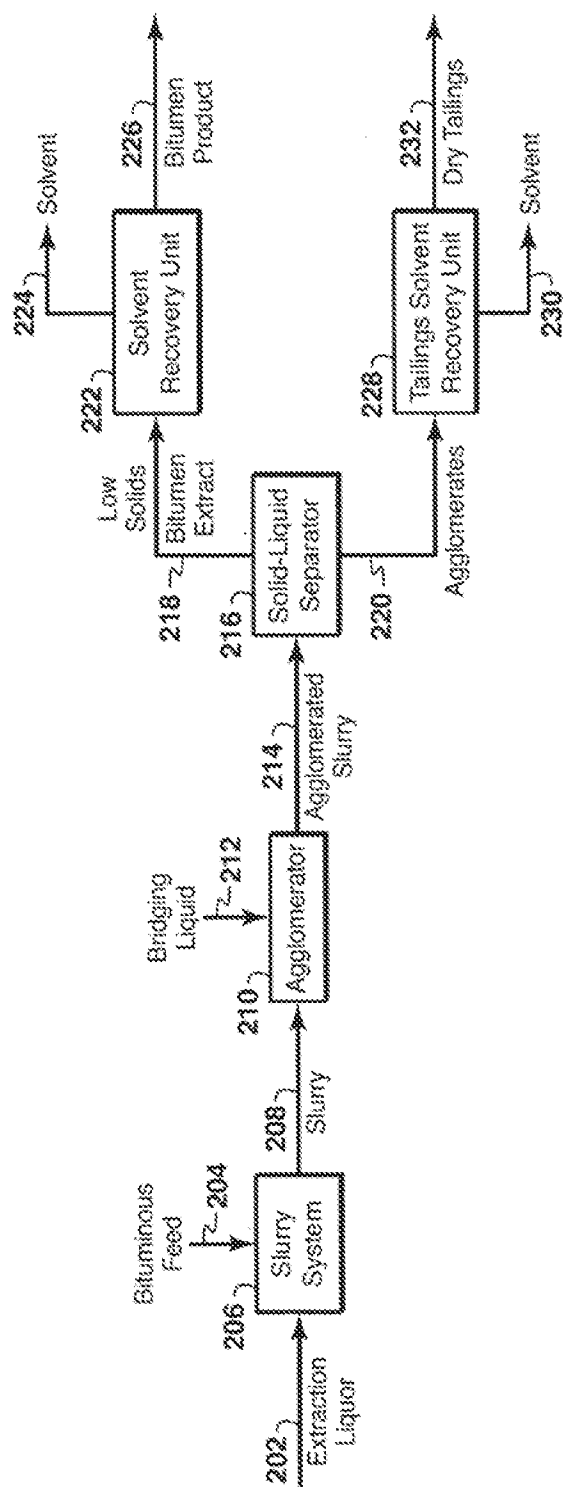


FIG. 2

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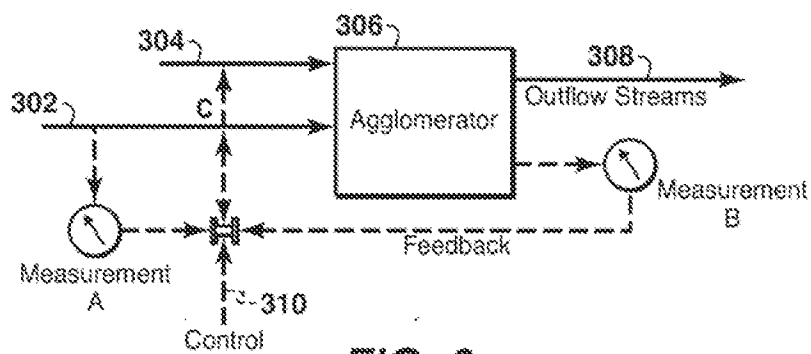


FIG. 3

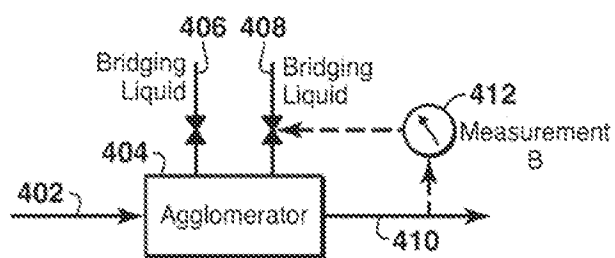


FIG. 4

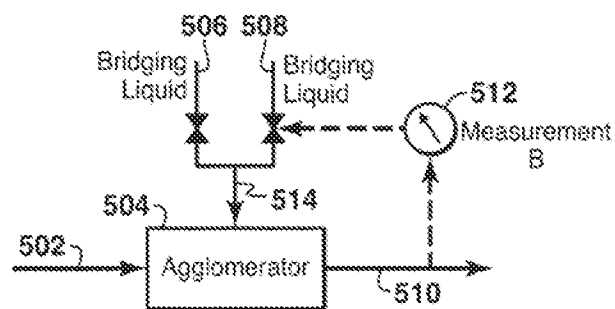
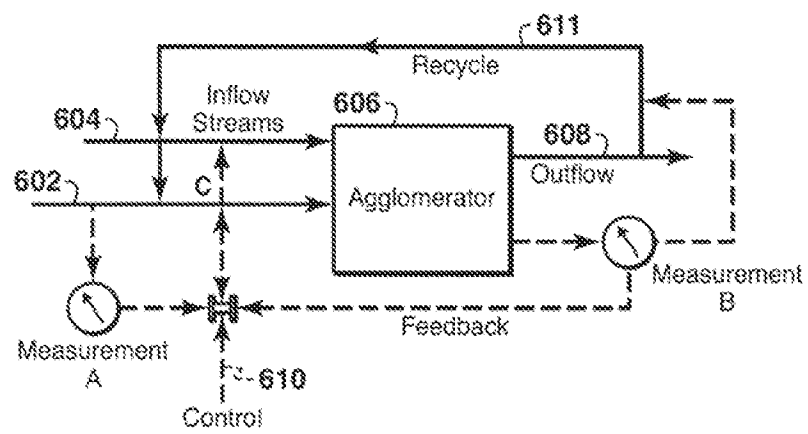


FIG. 5

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**FIG. 6**

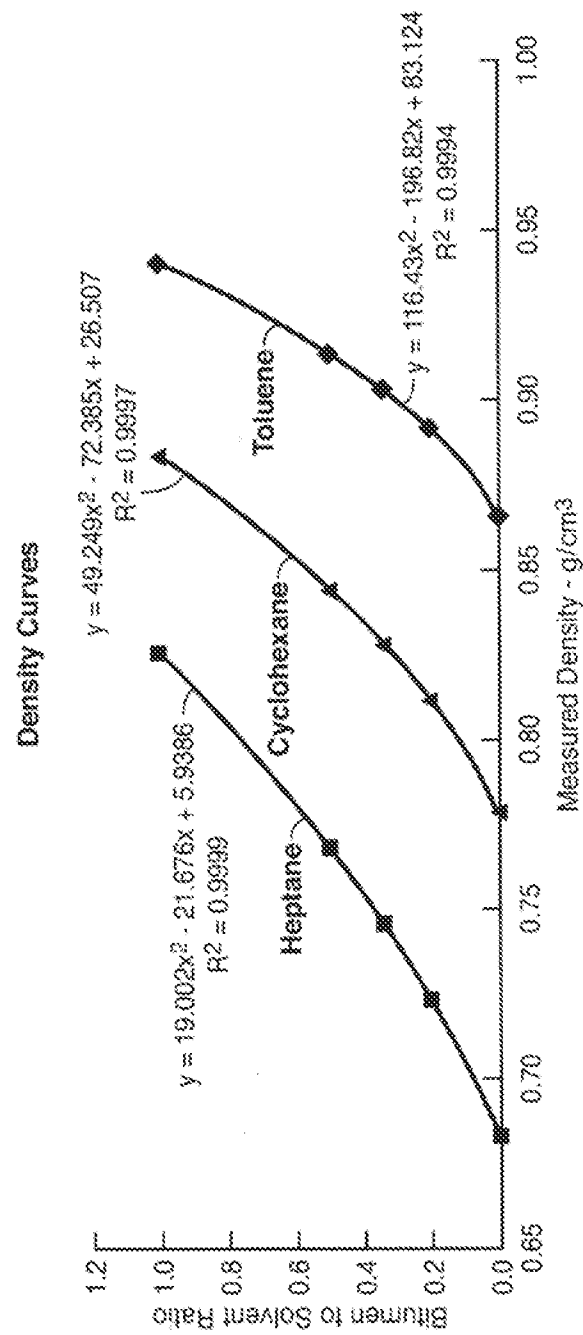


FIG. 7

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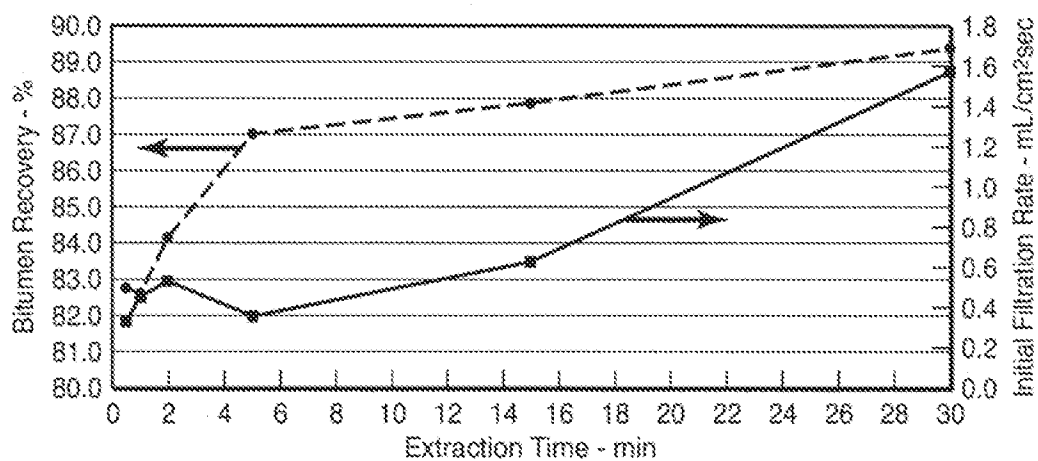


FIG. 8

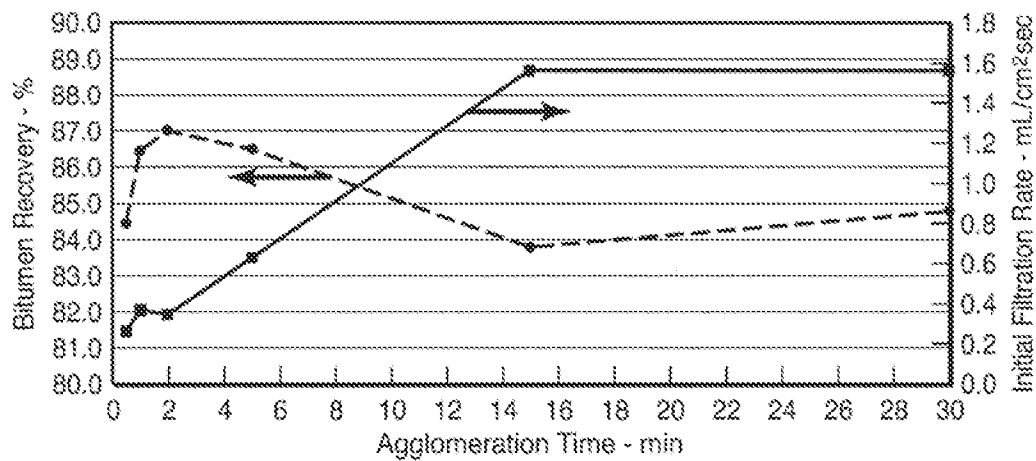


FIG. 9

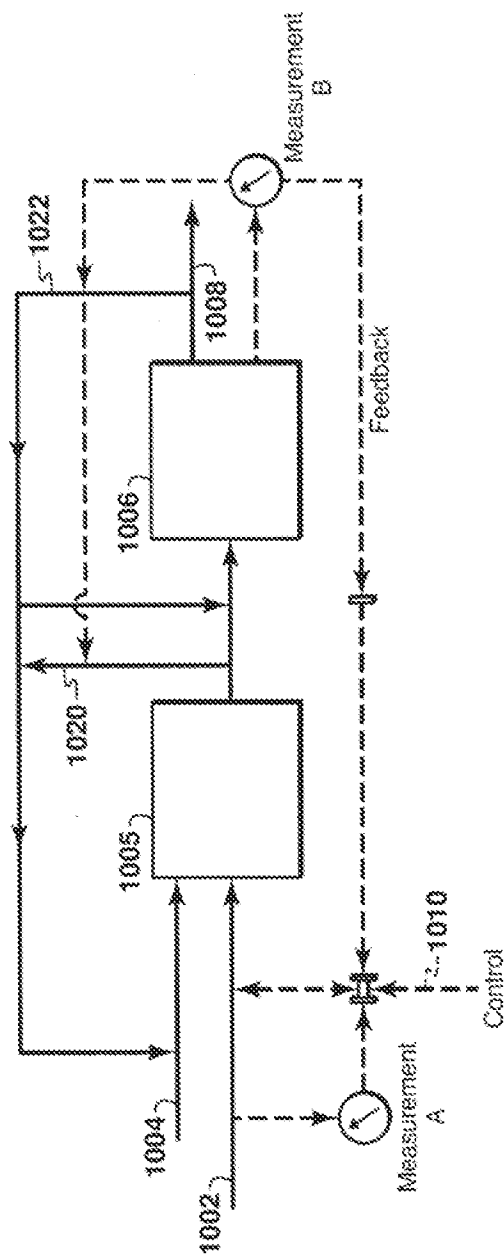


FIG. 10