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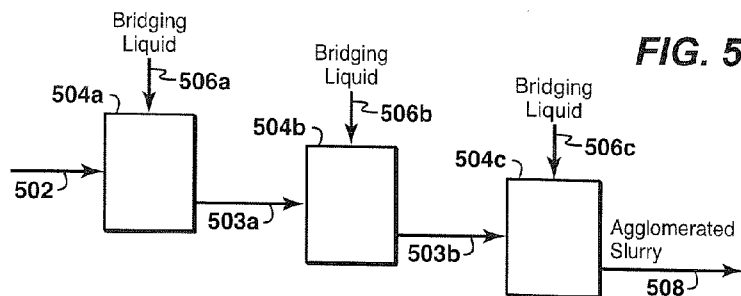
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(54) Title: METHOD OF PROCESSING A BITUMINOUS FEED BY STAGED ADDITION OF A BRIDGING LIQUID

**FIG. 5**

(57) Abstract: 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 in at least two stages and solids within the slurry are agitated to form an agglomerated slurry comprising agglomerated solids and a low solids bitumen extract. The agglomerates are then separated from the low solids bitumen extract. Potential benefits may include the production of smaller and more uniform agglomerates. The former may lead to higher bitumen recoveries and the latter may improve the solid-liquid separation rate. The bridging liquid may be added in an area of relatively high shear rates. Between stages of bridging liquid addition, agglomerates may be removed.

**METHOD OF PROCESSING A BITUMINOUS FEED BY
STAGED ADDITION OF A BRIDGING LIQUID**

CROSS REFERENCE TO RELATED APPLICATIONS

[0001] This application claims the benefit of Canadian patent application number
5 2,740,468 filed on May 18, 2011 entitled METHOD OF PROCESSING A BITUMINOUS FEED
BY STAGED ADDITION OF A BRIDGING LIQUID, the entirety of which is incorporated
herein.

FIELD

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

BACKGROUND

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

15 **[0004]** Solvent extraction processes for the recovery of the hydrocarbons have been
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
20 hydrocarbon extract. Solids agglomeration is a technique that can be used to deal with this
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
25 addition of a second liquid. The second liquid preferentially wets the solids but is immiscible
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
30 suspension liquid.

[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 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 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 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 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 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.

[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 with 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. The use of sludge, however, introduces other challenges such as the fact that the appropriate sludge may not be readily available at the mine site. Furthermore, the use of sludge as the bridging liquid leads to larger agglomerates that are more prone to entrapment of bitumen.

[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
5 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
10 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.

15 **[0013]** The micro-agglomeration process described in U.S. Patent No. 4,719,008 (Sparks et al.) requires careful control of the bridging 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
20 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 (Sparks et al.) 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.
25 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.

30 **[0014]** In previously described SESA processes, the bridging liquid is either added directly to the dry oil sands or it 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
35 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. 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
5 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.

[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
10 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 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.
15 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 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
20 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 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
25 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 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
30 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.
35 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] It would be desirable to provide an alternative or improved method for processing a bituminous feed.

SUMMARY

[0020] 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 in at least two stages and solids within the slurry are agitated to form an agglomerated slurry comprising agglomerated solids and a low solids bitumen extract. The bridging liquid is added to the slurry in regions having higher shear rates than a median shear rate within the slurry. The agglomerates are then separated from the low solids bitumen extract. Potential benefits may include the production of smaller and more uniform agglomerates. The former may lead to higher bitumen recoveries and the latter may improve the solid-liquid separation rate.

[0021] 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 in at least two stages and agitating solids within the slurry to form an agglomerated slurry comprising agglomerates and a low solids bitumen extract; said bridging liquid being added to the slurry in regions having higher shear rates than a median shear rate within the slurry; and c) separating the agglomerates from the low solids bitumen extract.

[0022] 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

[0023] Embodiments of the present disclosure will now be described, by way of example only, with reference to the attached Figures.

[0024] Fig. 1 is a flow chart illustrating a disclosed embodiment.

5 [0025] Fig. 2 is a schematic illustrating a disclosed embodiment.

[0026] Fig. 3 is a schematic illustrating a disclosed embodiment.

[0027] Fig. 4 is a schematic illustrating a disclosed embodiment.

[0028] Fig. 5 is a schematic illustrating a disclosed embodiment.

[0029] Fig. 6 is a schematic illustrating a disclosed embodiment.

10

DETAILED DESCRIPTION

[0030] The present disclosure relates to a method of processing a bituminous feed using staged addition of a bridging liquid. 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. 15 2,724,806 ("Adeyinka et al."), filed December 10, 2010 and entitled "Processes and Systems for Solvent Extraction of Bitumen from Oil Sands".

[0031] Prior to describing embodiments specifically related to the staged addition of bridging liquid, a summary of the processes described in Adeyinka et al. will now be provided.

20 *Summary of Processes of Solvent Extraction Described in Adeyinka et al.*

[0032] 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 25 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 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 30 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.

Staged Addition of Bridging liquid

[0033] 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 in at least two stages and solids within the slurry are agitated to form an agglomerated slurry comprising agglomerated solids and a low solids bitumen extract (**104**). The bridging liquid is added to the slurry in regions having higher shear rates than a median shear rate within the slurry. The agglomerates are then separated from the low solids bitumen extract (**106**). Potential benefits may include the production of smaller and more uniform agglomerates. The former may lead to higher bitumen recoveries and the latter may improve the solid-liquid separation rate.

[0034] 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.

[0035] 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.

[0036] Figure 2 is a schematic of a disclosed embodiment with additional steps including downstream solvent recovery. 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 (**210**) to assist agglomeration of the slurry. Some form of agitation is also used to assist agglomeration as described below.

[0037] 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**).

5 **[0038]** The following additional steps may also be performed. The low solids bitumen extract (**218**) 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**).

10 **[0039]** 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. The bridging liquid is then added to the slurry in order to agglomerate the fine solids within the slurry. The rate of agglomeration may be controlled by a balance among agitation, fines content of the slurry, and bridging liquid addition. In this embodiment, the bitumen is first extracted from the bituminous feed prior to agglomeration in order to prevent (or limit) the agglomeration
15 process from hampering the dissolution of bitumen into the extraction liquor.

[0040] 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 0.3mm in size.

20 **[0041]** Figure 3 illustrates an embodiment where the bridging liquid is added to an agglomeration vessel at multiple locations within the vessel. As illustrated, the bituminous feed (**302**) is added to an agglomerator (**304**). Bridging liquid (**306**) is added at multiple stages along the flow path of the slurry. For example, multiple bridging liquid inlet ports may be arranged sequentially along the agglomerator. The agglomerated slurry (**308**) is also shown.

25 **[0042]** After each stage of bridging liquid addition, the slurry may be well agitated so that the bridging liquid comes into contact with the solids within the slurry in order to form agglomerates. In one embodiment, the residence time between each stage of bridging liquid addition is sufficient to allow agglomeration of some of the fine particles within the slurry. The residence time between each stage of bridging liquid addition may be greater than 30
30 seconds.

[0043] After each stage of bridging liquid addition and the resulting formation of agglomerates, the formed agglomerates may be removed from the slurry. Exemplary methods for removing the agglomerates include gravity separation or screening within the agglomerator. Agglomerates that are larger than 1 mm are typically undesirable due to the

increased chance of bitumen entrapment within the large agglomerates. These large agglomerates that are removed from the agglomerator may be separately comminuted by various methods known in the art to obtain agglomerates of the preferred size. For example, the agglomerates may be comminuted within attrition scrubbers or rod mills.

5 **[0044]** In one embodiment, the bridging liquid is added to the slurry at relatively high agitation or mixing energy regions in order to improve the dispersion of the bridging liquid within the slurry. Therefore, the bridging liquid may be added to the slurry in regions having higher shear rates than a median shear rate within the slurry. An example of a region of high shear rate within an agglomerator is adjacent the propellers of a mixing vessel such as
10 an attrition scrubber. The propellers themselves may contain suitable injection ports designed for injecting bridging liquid, at high shear, into the slurry. In another example, the bridging liquid may be added to the slurry within the pumps used to transport the slurry.

[0045] The staged addition of bridging liquid may be used to assist in more uniformly agglomerating solids. The amount of bridging liquid added at each stage of bridging liquid
15 addition may be selected to obtain the desired agglomerate size.

[0046] Figure 4 illustrates an embodiment where the bridging liquid is added to an agglomeration vessel “continuously”. As illustrated, the bituminous feed (**402**) is added to an agglomerator (**404**). Bridging liquid (**406**) is added “continuously” along the flow path of the slurry. As used herein “continuously” means that the bridging liquid is added at stages
20 separated by residence times that are significantly shorter than the residence time needed for agglomerate formation. For examples, the residence times between each bridging liquid stage may be less than 15 seconds, or less than 5 seconds. The agglomerated slurry (**408**) is also shown.

[0047] Figure 5 illustrates an embodiment where the bridging liquid is added to
25 multiple agglomerators. As illustrated, the bituminous feed (**502**) is added to a first agglomerator (**504a**), to which bridging liquid (**506a**) is added. The slurry (**503a**) exists the first agglomerator (**504a**) and enters the second agglomerator (**504b**), to which bridging liquid (**506b**) is added. The slurry (**503b**) exists the second agglomerator (**504b**) and enters the third agglomerator (**504c**), to which bridging liquid (**506c**) is added. The agglomerated
30 slurry (**508**) is also shown. The use of multiple agglomerators allows for distinct stages of agglomeration to occur within each vessel. For example, one agglomerator may be used for the initial nucleation of agglomerate particles. A second agglomerator may be used to grow the agglomerates. A third agglomerator may be used for comminution of agglomerates. Since these stages of agglomeration may occur in separate vessels, the operator may have
35 a greater level of control of the processes.

[0048] Figure 6 illustrates an embodiment where agglomerates are removed from the slurry after they form and before the injection of additional bridging liquid. As illustrated, the bituminous feed (**602**) is added to a first agglomerator (**604x**), to which bridging liquid (**606x**) is added. The slurry (**610**) exists the first agglomerator (**604x**) and enters a solid-liquid separator (**612**) (examples of which are described below) separating agglomerates (**614**) from the reduced-solids slurry (**616**). The reduced-solids slurry (**616**) is fed into the second agglomerator (**604y**), to which bridging liquid (**606y**) is added. The agglomerated slurry (**608**) is also shown. More than two agglomerators and more than one solid-liquid separator could be used.

10 **[0049]** Exemplary methods for removing the agglomerates include, but are not limited to, gravity separators such as thickeners or enhanced gravity separators such as hydrocyclones. The agglomerates may be removed from the slurry in order to reduce their additional growth. It has been shown in previous studies that in order to maximize bitumen recovery, it is desirable to keep the agglomerates average particle size to as low a value as possible commensurate with achieving economically viable solid liquid separation. If the agglomerates were to remain within the slurry, after subsequent bridging liquid additions, unagglomerated fine particles would preferentially attach to the agglomerates, thus increasing the chances of bitumen entrapment within the growing agglomerates. Additionally, a portion of the agglomerates that are removed from the agglomerators may be separately
15 comminuted by various methods known in the art to obtain agglomerates of the preferred size. For example, the agglomerates may be comminuted within attrition scrubbers or rod mills.

[0050] The agglomeration processes herein described may be used for the formation of macro-agglomerates or micro-agglomerates from the solids of the bituminous feed.
25 Macro-agglomerates are agglomerates that are predominantly greater than 2 mm in diameter. These agglomerates comprise both the fine particles (less than 44 μm) and sand grains of the oil sands. Micro-agglomerates are agglomerates that are predominately less than 1 mm in diameter and they principally comprise fine particles of the oil sands. It has been found that for the SESA process described above, the formation of micro-agglomerates
30 are more suitable for maximizing bitumen recovery for a range of oil sands grades.

Agitation

[0051] 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 bridging liquid
35 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.

Extraction Liquor

[0052] The extraction liquor comprises a solvent used to extract bitumen from the bituminous feed. The term "solvent" as used herein should be understood to mean either a single solvent, or a combination of solvents.

10 **[0053]** 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 liquor in order to increase the volume of the extraction liquor without an increase in the
15 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.

[0054] The extraction liquor may be mixed with the bituminous feed to form a slurry where most or all of the bitumen from the oil sands is dissolved into the extraction liquor. In
20 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 vessels, 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 lower solids content may be more suitable.

25 **[0055]** 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.

[0056] While it is not necessary to use a low boiling point solvent, when it is used,
30 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.

[0057] 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 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
5 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-heptane, hexane, iso-hexane, pentane, iso-pentane, or a combination thereof. Should a
10 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.

[0058] Exemplary cycloalkanes include cyclohexane, cyclopentane, or a mixture thereof.

[0059] If the solvent is selected as the distillate from a fractionation unit, it may for
15 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.

[0060] A mixture of C₄-C₁₀ cyclic and/or open chain aliphatic solvents would also be appropriate. For example, it can be a mixture of C₄-C₉ cyclic aliphatic hydrocarbons and paraffinic solvents where the percentage of the cyclic aliphatic hydrocarbon in the mixture is
20 greater than 50%.

[0061] 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 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.

[0062] The solvent may also include additives. These additives may or may not be 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 included cements, fly ash,
30 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 removal of suspended solids and prevent emulsification of the two solvents. Exemplary
35 additives include methanoic acid, ethylcellulose and polyoxyalkylate block polymers.

Bridging Liquid

- [0063]** 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 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 solution 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.
- 5
- [0064]** The total amount of bridging liquid added to the slurry may be such that a ratio of bridging liquid to solids within the agglomerated slurry is in the range of 0.02 to 0.25, or 0.05 and 0.11. The amount of bridging liquid that makes up this ratio includes the bridging liquid added to the slurry and the connate water from the bituminous feed. The amount of bridging liquid that is added at a stage may be the same for each stage, or may be different.
- 10
- [0065]** 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 than 40 wt.%. In one embodiment, the agglomerated slurry has a solids content of 20 to 70 wt.%.
- 15
- [0066]** The composition of the bridging liquid, for example salinity and/or fines content, may be the same or different depending on which stage along the slurry flow path the bridging liquid is added. Additionally, the amount of the bridging liquid that is added to the slurry at each stage of bridging liquid addition may be the same or different. For example, the bridging liquid added during a first stage may have a salinity that is at least 10% higher or lower than a salinity of a bridging liquid added during a second, subsequent stage. In another examples, the bridging liquid added during a first stage may have a suspended solids content that is at least 10 % higher or lower than a suspended solids content of a bridging liquid added during a second, subsequent stage.
- 20
- 25

General Experimental Observations

- [0067]** Preliminary batch tests of solvent extraction have shown that bitumen recovery increased by as much as five percentage points when the bridging liquid was added into the process vessel at intermittent time intervals during the agglomeration process compared to the case where all the bridging liquid was added at the beginning of the agglomeration process. The improved bitumen recovery performance demonstrated by the
- 30

staged bridging liquid addition is also supported by observations made during batch testing of the solids agglomeration process within a mixing vessel. In these tests, a translucent organic fluid was used as the continuous phase with sand and clay as the solids, and water as the bridging liquid. When the water was added all at once to the slurry comprising the organic fluid and solids, large agglomerates quickly formed and began to segregate from the slurry. The slurry remained segregated until sufficient mixing energy was applied to the slurry to break up the initially formed agglomerates and disperse the water. The particle size distribution of the agglomerates formed in this suboptimal case was found to be broad with a significant amount of agglomerates being outside the size range for optimal bitumen recovery and solid-liquid separation. For the tests where the water was added gradually and near the mixing impellers for increased mixing energy, the water rapidly dispersed and minimal segregation of agglomerates was observed. The particles size distribution of these agglomerates was found to be narrow, and as a result the bridging liquid can be used more efficiently to obtain the desired agglomerate size.

15 *Ratio of Solvent to Bitumen for Agglomeration*

[0068] 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.

[0069] 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.

Slurry System

35 **[0070]** 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.

Solid-Liquid Separator

5 **[0071]** 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, a clarifier, a cyclone, a screen, a belt filter or a combination thereof.

10 **[0072]** 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 solid-liquid separator is employed together with a secondary solid-liquid separator. When
15 primary and secondary units are both employed, generally, the primary unit separates agglomerates, while the secondary unit involves washing agglomerates.

[0073] 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 December 10, 2010.

20 *Secondary Stage of Solid-Liquid Separation to Wash Agglomerates*

[0074] 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 separator, while the secondary stage may occur within the primary unit,
25 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 agglomerates, the less clean will be the solvent used to wash agglomerates
30 at that stage. The result being that the cleanest wash of agglomerates is conducted using the cleanest solvent.

[0075] 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 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 conventional fractionation tower or a distillation unit.

- 5 **[0076]** 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.

- 10 **[0077]** 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.

Recycle and Recovery of Solvent

- 15 **[0078]** The process may involve removal and recovery of solvent used in the process.

- 20 **[0079]** 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. 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 have 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.

- 25 **[0080]** 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.

- 30 **[0081]** 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.

- [0082]** 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 the agglomerator that avoids precipitation of asphaltenes.

Extraction Step may be Separate from Agglomeration Step

- 5 **[0083]** Solvent extraction may be conducted separately from agglomeration in certain embodiments of the process. Unlike certain prior processes, where the solvent is first exposed to the bituminous feed within the agglomerator, certain embodiments described herein include contact of the extraction liquor with the bituminous feed prior to the agglomeration step. This has the effect of reducing residence time in the agglomerator, when compared to certain previously proposed processes which require extraction of bitumen and agglomeration to occur simultaneously. The instant process is tantamount to agglomeration of pre-blended slurry in which extraction via bitumen dissolution is substantially or completely achieved separately. Performing extraction upstream of the agglomerator permits the use of enhanced material handling schemes whereby flow/mixing systems such as pumps, mix boxes or other types of conditioning systems can be employed. Additionally, performing extraction upstream of the agglomerator prevents the agglomeration process from hampering the dissolution of bitumen into the extraction liquor.

- 20 **[0084]** Because the extraction may occur upstream of the agglomeration step, the residence time in the agglomerator may be reduced. One other reason for this reduction is that by adding components, such as water, some initial nucleation of particles that ultimately form larger agglomerates can occur prior to the agglomerator.

Dilution of Agglomerator Discharge to Improve Product Quality

- 25 **[0085]** 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 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.

- 30 **[0086]** When dilution of agglomerator discharge is employed in this embodiment, the 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 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.

5 *Potential Advantages*

[0087] There may be advantages of embodiments described herein, for instance as compared to SESA. It is believed that adding the bridging liquid at multiple stages along the flow path of oil sands slurry can lead to an agglomeration process that is more controllable and yield agglomerates of more uniform size. It is also believed that adding the bridging liquid at multiple stages along the flow path of oil sands slurry will reduce the overall energy requirement of the agglomeration process. Similar advantages have been realized in the agglomeration of coal fine particles (see U.S. Patent No. 4,153,419).

[0088] The high energy requirements of solids agglomeration process is a well known limitation. Embodiments described herein are expected to reduce the total energy needed to form the agglomerates. The reduction in energy is due to the lower power requirement needed for the agglomeration process. The reduction in power, in turn, is due to the staged addition of bridging liquid. Since the bridging liquid is added in stages, the viscosity of the oil sands does not increase as much as it would if all the bridging liquid was added at once. A reduction in the power requirement means that that the torque requirements of motors used in certain types of agglomeration vessel can be reduced. In the case of rotating type vessels, the required amount of milling can be reduced. Furthermore, the wear of the internals of the vessels will be dramatically reduced due to a reduction in the required mixing intensity.

[0089] Without intending to be bound by theory, it is believed that staged addition of bridging liquid helps balance the rate of agglomeration with the rate of mixing. In the presence of a significant amount of bridging liquid, the agglomeration of the solids occur at a rate that is much more rapid than the rate of mixing of the slurry. In fact, the agglomeration process itself tends to hamper mixing by increasing the effective viscosity of the slurry. The staged addition of the bridging liquid may modulate the rate of agglomeration at any particular location in the vessel. Thus, the mixing of the slurry can match the agglomeration process to ensure that the slurry remains relatively homogeneous. The addition of bridging liquid in high mixing energy regions of the slurry assist bridging liquid dispersion throughout the slurry. Additionally, removing the agglomerates from the slurry after each stage of bridging liquid addition reduces the viscosity of the slurry and prevents (or limits) excessive growth of the formed agglomerates.

[0090] Another potential advantage of certain embodiments is the shifting of the growth of agglomerates to a layering mechanism rather than a coalescence mechanism. The layering mechanism refers to agglomerate growth where the individual fine particles stick on the surface of already formed agglomerates. The coalescence mechanism refers to agglomerate growth where two or more agglomerates stick together. The layering mechanism should result in more compact agglomerates with less bitumen extract entrapped therein. In the cases where the formed agglomerates remain in the slurry, these agglomerates act as seed particles and shift the agglomeration process to more of a layering mechanism than a coalescence mechanism, which may dominate the agglomerate growth mechanism if all the bridging liquid was introduced in a single stage.

[0091] In the preceding description, for purposes of explanation, numerous details 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.

[0092] 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.

Batch Experiments

[0093] Experiments were conducted to test the effectiveness of using staged addition of bridging liquid in order to agglomerate oil sand solids within a slurry. 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 agglomeration process. The agglomerates were also visually inspected for their size and uniformity.

[0094] 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 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.

[0095] 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 the experiments. The agglomeration experiments were conducted at room temperature (22 °C).

[0096] 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 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.

[0097] 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.

Experiment 1: agglomeration by adding all the bridging liquid at one stage

[0098] 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 homogenize the mixture and to extract the bitumen that was in the oil sands. After 5 minutes of mixing, 16.8g of water was quickly poured into the vessel through a sample port. The mixture was then mixed at 1500 rpm for an additional 2 minutes to agglomerate the solids.

[0099] 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. The initial drainage rate for solids agglomerated by adding all the bridging liquid at one stage to the solids slurry was 0.35 $\text{mL}/(\text{cm}^2\text{sec})$.

[0100] 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. The bitumen recovery from this solvent extraction with solids agglomeration process was 87 %.

5 *Experiment 2: agglomeration by adding all the bridging liquid at one stage*

[0101] The same conditions as Experiment 1 was repeated with the agglomeration time extended to 15 minutes instead of 2 minutes. The initial drainage rate for solids agglomerated by adding all the bridging liquid at one stage and extending the agglomeration time increased to 1.6 mL/(cm²sec). However, the bitumen recovery for this extraction
10 process dropped to 83.8%.

Experiment 3: agglomeration by using staged addition of bridging liquid to solids slurry

[0102] 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
15 minute of mixing, 5.6 g of water was poured into the vessel through a sample port and the mixture was mixed for 30 seconds. Subsequently, 5.6 g of water was poured into the vessel and the mixture was mixed for an additional 30 seconds. Lastly, 5.6 g of water was poured into the vessel and the mixture was mixed for 1 minutes. All mixing was done at room temperature.

20 **[0103]** 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 cm². The solids bed height was 10.8 cm. A portion of
25 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. The initial drainage rate for solids agglomerated by adding bridging liquid in three separate stages to the solids slurry was 1.04 mL/(cm²sec).

30 **[0104]** The remaining supernatant was poured onto the solid bed and allowed to filter though. 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. The bitumen recovery

from this solvent extraction with solids agglomeration process was 86.3 %.

[0105] The drainage rate of the agglomerates formed by using staged addition of the bridging liquid was approximately 3 times greater than that of agglomerates formed when all the bridging liquid is added in one stage (compare Experiment 3 to Experiment 1). The drainage rate of the single stage agglomeration process can be increased by extending the agglomeration time (see Experiment 2) in order to form larger agglomerates. However, the larger agglomerates results in a reduction in the bitumen recovery. In contrast, the staged addition of bridging liquid resulted in an increase in drainage rate without a significant reduction in bitumen recovery. Visual inspection of Experiment 3 agglomerates did not reveal significantly larger agglomerates compared to the agglomerates of Experiment 1. This suggests that the faster drainage rate of the agglomerates formed by staged addition of bridging liquid is due to more uniform agglomerates rather than larger agglomerates.

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 in at least two stages and agitating solids within the slurry to form an agglomerated slurry comprising agglomerates and a low solids bitumen extract; said bridging liquid being added to the slurry in regions having higher shear rates than a median shear rate within the slurry; and
10 c) separating the agglomerates from the low solids bitumen extract.
2. The method of claim 1, wherein the regions having higher shear rates than a median shear rate within the slurry are regions adjacent propellers used to agitate the slurry.
3. The method of claim 2, wherein the propellers comprise ports for adding the bridging liquid to the slurry.
- 15 4. The method of claim 1, wherein the bridging liquid is added in at least two stages in a single agglomerator.
5. The method of claim 1, wherein the bridging liquid is added continuously or intermittently in one or more agglomerators.
6. The method of claim 1, wherein the bridging liquid is added in at least two
20 agglomerators.
7. The method of claim 1, wherein step b) comprises:
 - i) adding a first portion of the bridging liquid to the slurry;
 - ii) agitating solids within the slurry to form agglomerates;
 - iii) removing agglomerates from the slurry to form a solids-reduced slurry;
 - 25 iv) adding a second portion of the bridging liquid to the solids-reduced slurry; and
 - v) agitating solids within the solids-reduced slurry to form agglomerates.
8. The method of claim 7, wherein steps i) to v) are performed at least twice.
9. The method of claim 7, wherein the first portion of the bridging liquid is added to a first agglomerator, the second portion of the bridging liquid is added to a second
30 agglomerator, and agglomerates are removed to form the solids-reduced slurry using a solid-liquid separator.

10. The method of claim 7, further comprising comminuting removed agglomerates of step iii).
11. The method of claim 10, wherein the comminuting is effected in an attrition scrubber.
12. The method of claim 10, wherein the comminuting is effected in a mill.
- 5 13. The method of claim 1, wherein bridging liquid added during a first stage has a salinity that is at least 10% higher or lower than a salinity of a bridging liquid added during a second, subsequent stage.
14. The method of claim 1, wherein bridging liquid added during a first stage has a suspended solids content that is at least 10 % higher or lower than a suspended solids
10 content of a bridging liquid added during a second, subsequent stage.
15. The method of claim 1, wherein the bridging liquid is added in at least three stages.
16. The method of claim 1, further comprising recovering the solvent from the low solids bitumen extract to form a bitumen product.
17. The method of claim 16, further comprising washing the agglomerates of step c) with
15 the solvent recovered from the low solids bitumen extract.
18. The method of claim 1, further comprising washing the agglomerates of step c) with a solvent, which solvent is the same as or different from the solvent of step a), to extract additional bitumen and to form washed agglomerates.
19. The method of claim 1, further comprising recovering solvent from the agglomerates,
20 which have been separated from the low solids bitumen extract.
20. The method of claim 18, further comprising recovering solvent from the washed agglomerates.
21. 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%.
- 25 22. The method of claim 1, further comprising, prior to step a), contacting the bituminous feed with additional extraction liquor to begin extraction.
23. The method of claim 1, wherein the bridging liquid is water.
24. The method of claim 1, wherein the bridging liquid is an aqueous solution.
25. The method of claim 1, wherein at least 80 wt. % of the agglomerates of step c) are
30 between 0.1 and 1 mm.
26. The method of claim 1, wherein the agglomerated slurry has a solids content of 20 to

70 wt%.

27. The method of claim 1, wherein the solvent comprises an organic solvent or a mixture of organic solvents.

28. The method of claim 27, wherein the solvent comprises a paraffinic solvent, a cyclic
5 aliphatic hydrocarbon, or a mixture thereof.

29. The method of claim 28, wherein the paraffinic solvent comprises an alkane, a natural gas condensate, a distillate from a fractionation unit, or a combination thereof containing more than 40% small chain paraffins of 5 to 10 carbon atoms.

30. The method of claim 29, wherein the alkane comprises a normal alkane, an
10 isoalkane, or a combination thereof.

31. The method of claim 29, wherein the alkane comprises heptane, iso-heptane, hexane, iso-hexane, pentane, iso-pentane, or a combination thereof.

32. The method of claim 28, wherein the cyclic aliphatic hydrocarbon comprises a cycloalkane of 4 to 9 carbon atoms.

15 33. The method of claim 32, where the cycloalkane comprises cyclohexane, cyclopentane, or a mixture thereof.

34. The method of claim 1, wherein the solvent comprises at least 50 wt. % cyclohexane.

35. The method of claim 1, wherein the extraction liquor comprises residual solids.

36. The method of claim 1, wherein the bridging liquid comprises solid fines.

20 37. The method of claim 36, wherein bridging liquid has a solids content of less than 40 wt% solid fines.

38. The method of claim 1, wherein a ratio of the solvent to bitumen in the agglomerated slurry is less than 2:1.

39. The method of claim 1, wherein step b) comprises agitating by mixing, shaking, or
25 rolling.

40. The method of claim 1, wherein the bituminous feed is derived from oil sands.

41. The method of claim 1, wherein 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 to 0.25.

42. The method of claim 1, wherein a ratio of bridging liquid plus connate water from

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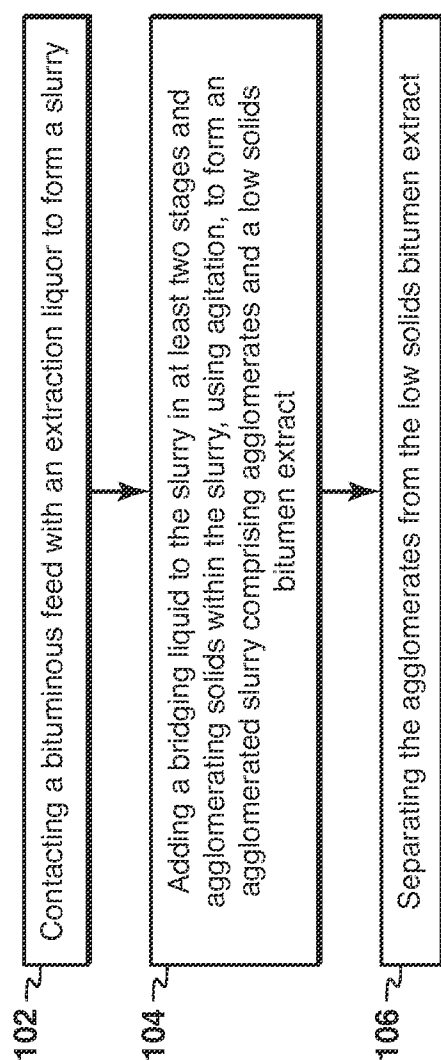


FIG. 1

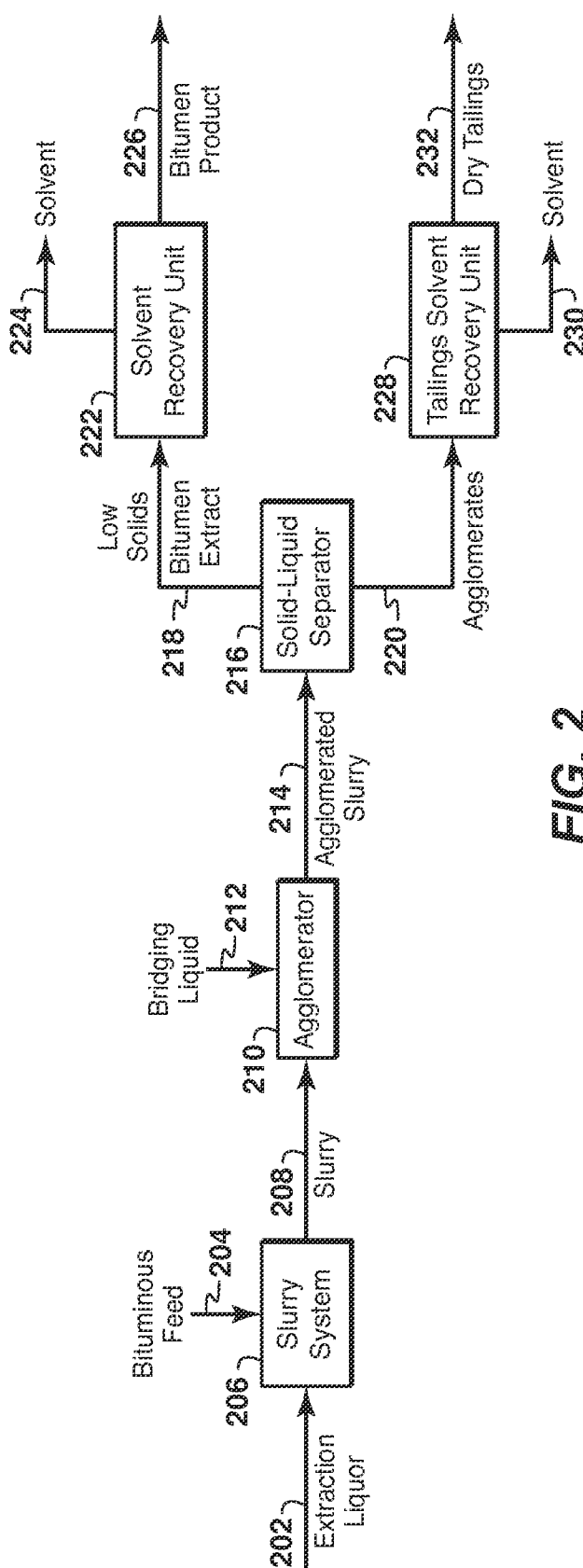
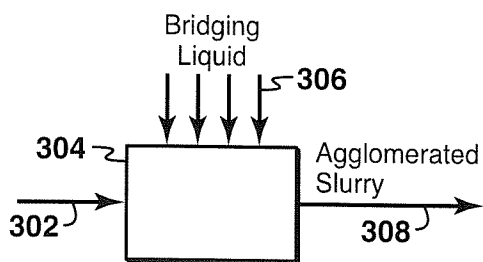
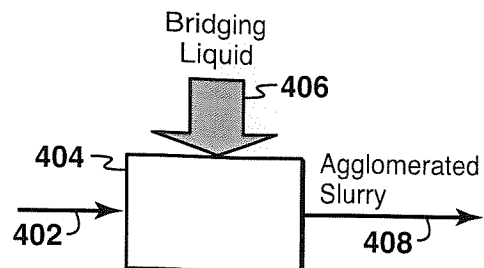
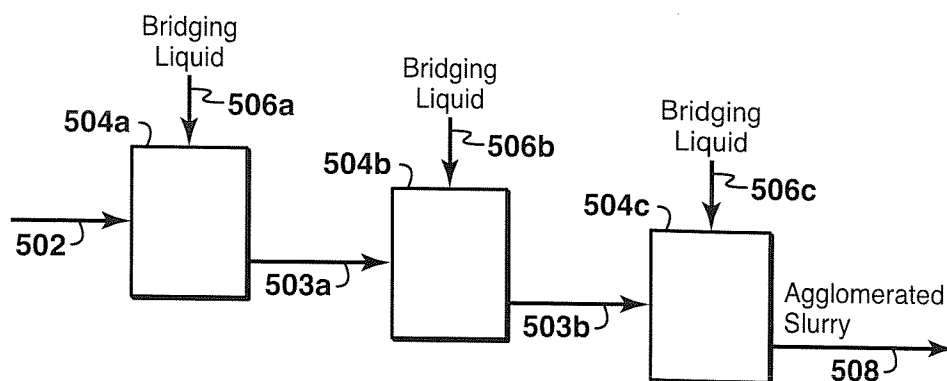
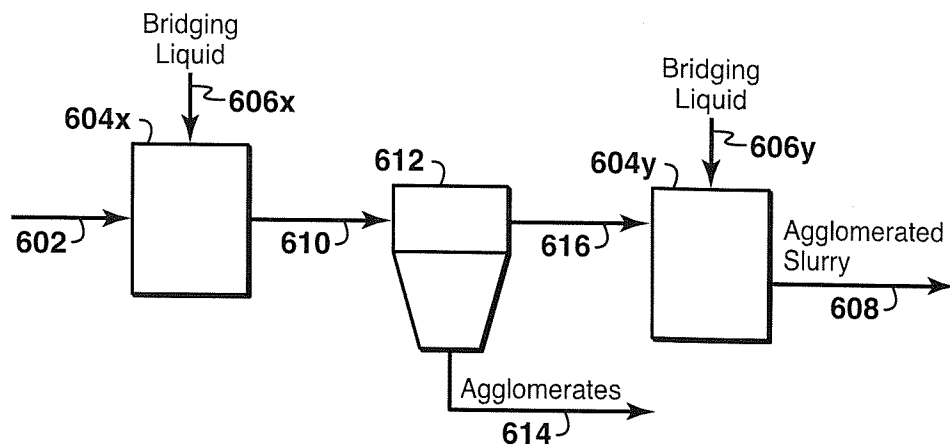


FIG. 2

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**FIG. 3****FIG. 4****FIG. 5****FIG. 6**

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US 12/28565

A. CLASSIFICATION OF SUBJECT MATTER

IPC(8) - C10G 1/04 (2012.01)

USPC - 208/390

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC(8) -- C10G 1/04 (2012.01)

USPC -- 208/390

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

IPC(8) -- C10G 1/04; C01G (2012.01)

USPC -- 208/390, 210/512, 210/513

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

PubWest (PGPB,USPT,USOC,EPAB,JPAB); DialogWeb (File 348 European Patents Fulltext; File 349 WIPO/PCT Patents Fulltext); USPTO; Espacenet; Google Patents; Google Scholar -- AGGLOMER\$ AQUEOUS BITUMEN\$ CYCLOHEXANE FINES HEXANE JET-MIXER LIQUID JET MICRON OIL PENTANE SANDS SLURR\$ SOLVENT WATER

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X --- Y	US 3,984,287 A (Meadus et al.) 05 October 1976 (05.10.1976) Fig 1; col 4, ln 6-9; col 4, ln 12-28; col 4, ln 40-54; col 4, ln 55-61; col 4, ln 61-62	1,5,7,8,13-15,23,24, 27,39,40 ----- 2-4,6,9-12,16-22,25,26, 28-38,41,42
Y	US 4,719,008 A (Sparks et al.) 12 January 1988 (12.01.1988) Fig 2; Fig 3; col 4, ln 50 to col 5, ln 2; col 5, ln 31-45; col 9, ln 34-45	10-12, 32-37
Y	US 2009/0294332 A1 (Ryu) 03 December 2009 (03.12.2009) Fig 1; para [0009]; [0011]; [0045]; [0047]; [0049]; [0050]; [0063]; [0066]	2-4,6,9,16,17
Y	WO 2011/021092 A2 (Filby et al.) 24 February 2011 (24.02.2011) Fig 1; page 3, ln 22-24; page 6, ln 17-25; page 11, ln 30 to page 12, ln 2; page 12, ln 23-26; page 14, ln 23-25; page 17, ln 10-17; page 23, ln 21 to page 24, ln 11	17-21,25,26,28-33, 35-38,41,42
Y	US 4,424,112 A (Rendall) 03 January 1984 (03.01.1984) Fig 1; Fig 2B; col 7, ln 38-68; col 8, ln 58 to col 9, ln 28	22
Y	US 4,544,477 A (Taylor) 01 October 1985 (01.10.1985) Fig 1; Fig 5; Fig 14; col 8, ln 2-13; col 12, ln 3-9; col 12, ln 54-65; col 15, ln 49 to col 16, ln 18	2-4,6,9



Further documents are listed in the continuation of Box C.



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"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search

05 JUNE 2012 (05.06.2012)

Date of mailing of the international search report

22 JUN 2012

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PCT OSP: 571-272-7774

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US 12/28565

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 2008/0169222 A1 (Ophus) 17 July 2008 (17.07.2008) Fig 1; Table 1; para [0008]; [0050]; [0052]; [0054]; [0070]	1-42
A	US 3,542,666 A (Simpson) 24 November 1970 (24.11.1970) Figure; col 5, ln 12-65	1-42
A	US 5,017,281 A (Sadeghi et al.) 21 May 1991 (21.05.1991) Fig 1; col 7, ln 1-10	1-42
A	US 5,143,598 A (Graham et al.) 01 September 1992 (01.09.1992) Fig 1; Fig 2	1-42