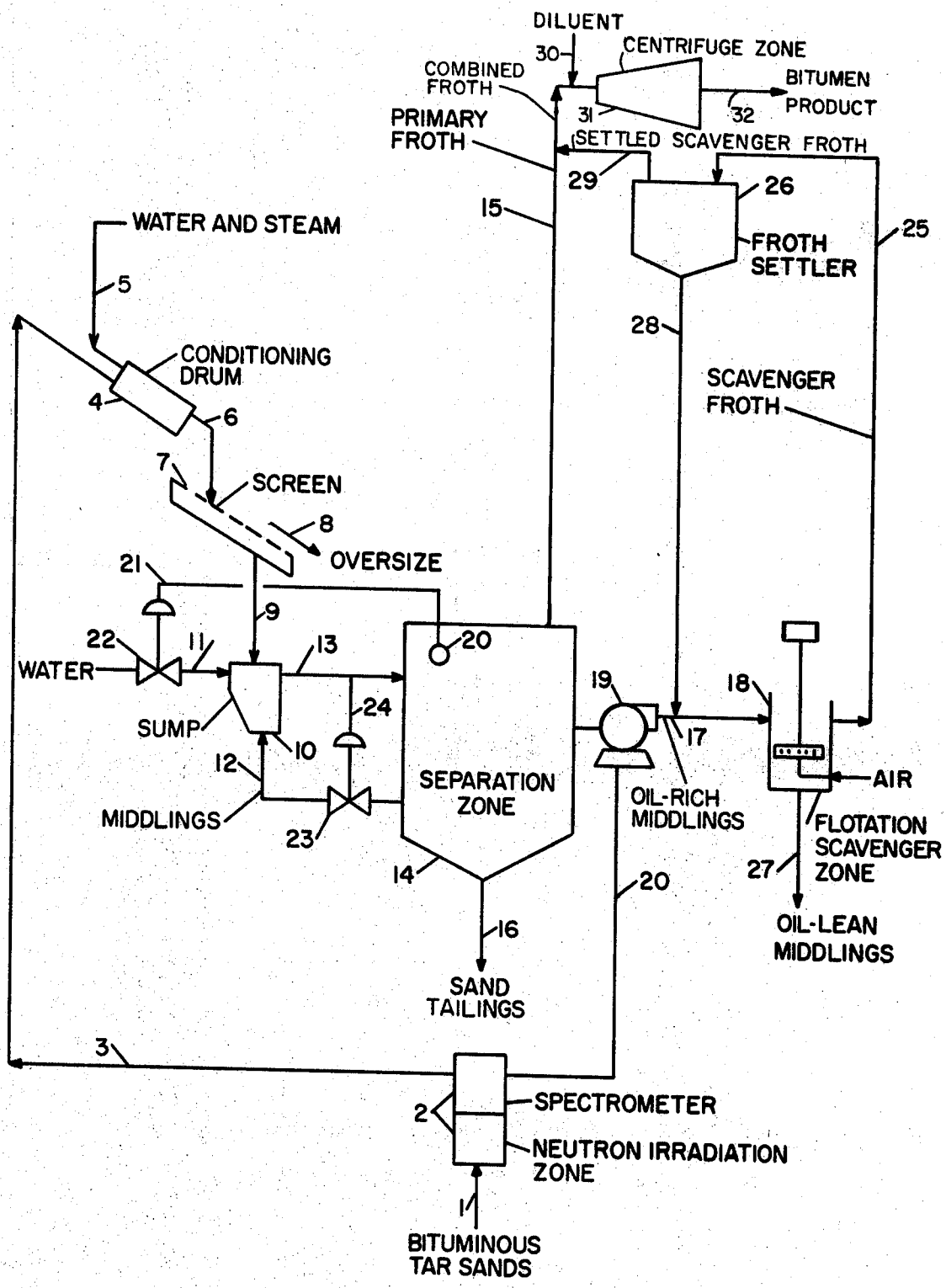
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HOT WATER PROCESS

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HOT WATER PROCESS

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ABSTRACT OF THE DISCLOSURE

The specification discloses a method of determining the processability of bituminous tar sands and regulating the amount of water mixed with tar sands in a hot water process according to the determined ease of processability of the sands. By the invention the aluminum content of the sands is determined and related to the clay content of the sands. The sands clay content is used as a variable for determining the amount of water and reagent to be mixed with the sands in the hot water process.

This invention relates to a method for determining the processability of bituminous tar sands. Specifically the invention relates to an improved hot water process for separating bitumin from tar sands.

Deposits of tar sands exist at various places throughout the world. The most extensive deposits are found in northern Alberta, Canada. The deposits are of Mesozoic Age. They underlie truncated Paleosoic limestones and are usually overlain by Pleistocene sediments. The deposits underlay more than 13,000 square miles at depths of 0 to 2000 feet. Total recoverable reserves of oil from the deposits after extraction and processing are estimated at more than 300 billion barrels.

The sands are composed of a siliceous material, generally having a size greater than that passing a 325 mesh screen, saturated with a relatively heavy, viscous bitumen in quantities of from 5 to 21 weight percent of the total composition. More typically, the bitumen content of the sands is about 8 to 15 percent. This bitumen is quite viscous and contains typically 4.5 percent sulfur and 38 percent aromatics. Its specific gravity at 60° F. ranges typically from about 1.00 to about 1.06. The tar sands also contain clay and silt in quantities of from 1 to 50 weight percent of the total composition. Silt is normally defined as mineral which will pass a 325 mesh screen but which is larger than 2 microns. Clay is mineral smaller than 2 microns including some siliceous material of that size.

There are several well-known processes for effecting the separation of bitumen from the tar sands. In the so-called "cold water" method, the separation is accomplished by mixing the sands with a solvent capable of dissolving the bitumen constituent. The mixture is then introduced into a large volume of water, water with a surface agent added, or a solution of a neutral salt in water. The combined mass is then subjected to a pressure or gravity separation.

In the hot water method, the bituminous sands are jetted with steam and mulled with a minor amount of hot water at temperatures in the range of 140° F. to 210° F. The resulting pulp is dropped into a stream of circulating hot water and carried to a separation cell maintained at a temperature of about 150° to 200° F. In the separation cell, sand settles to the bottom as tailings and bitumen rises to the top in the form of a bitumen froth. An aqueous middlings layer containing some mineral and bitumen is formed between these layers. A scavenger step may be conducted on the middlings layer from the primary separation step to recover additional amounts of bitumen therefrom. This step usually comprises aerating

the middlings as taught by K. A. Clark, "The Hot Water Washing Method," Canadian Oil and Gas Industries 3, 46 (1950). The froths can be combined, diluted with naphtha and centrifuged to remove more water and residual mineral. The naphtha is then distilled off and the bitumen is coked to a high quality crude suitable for further processing. The hot water process is described in detail in United States patent application Ser. No. 509,589, Floyd et al., now Pat. No. 3,401,110.

Several techniques can be used for the mining of the tar sands. Deposits at or near the surface can be mined by open-pit mining techniques in which overburden, if any, is stripped away and the exposed sands mined by conventional digging equipment. Preferably the mining is by giant bucketwheel excavators which have been found to be extremely useful in this application. The mined material is then transferred to the processing area for separation of the bitumen.

The ease with which the bitumen is separated from the tar sands varies considerably through a deposit. Some sands result in high yields while others, from the same deposit, give low primary bitumen froth yields in the hot water process. This ease of processability is related to the fines content of the tar sands as described by Floyd et al. in United States patent application 509,589. Floyd et al. point out that if clay and silt are allowed to build up in the separation cell of the hot water process, the viscosity of the cell middlings layer will increase. Concurrently with such increase, an increase in the proportions of both bitumen and sand retained by the middlings will occur. If the clay and silt content is allowed to build up too high in the system, effective separation will no longer occur and the process will become inoperative. Floyd et al, propose regulating the recycling and withdrawal of middlings and input of fresh water to avoid inoperativeness. In Floyd et al., middlings viscosity and/or density is measured and input of fresh water determined thereby. It is proposed by the present invention to determine the fines content of the tar sands to their introduction to the hot water process and to regulate the fresh water input into the process according to the measured fines content of the sands so as to provide a constant anticipatory control on middlings viscosity. By the present invention the middlings viscosity can be controlled and maintained in the desired range of about 0.4 to 5.7 centipoise.

United States Pat. 3,273,967, Wilson, teaches a method for determining the processability of tar sands which comprises analyzing samples for their content of a member selected from the group consisting of iron and zinc, and correlating the analyses with the sample to determine the location of the more easily processable tar sand. The patent teaches that zinc and iron and particularly iron are the only elements which appear to vary to any extent so as to give an indication of the processability of the tar sands. In contrast to this teaching the present invention measures the aluminum content of the sands to determine their processability. It has been found that the aluminum content of the sands is a more accurate indicator of processability than the iron or zinc content—the more easily processable sands having the lower aluminum contents and the more difficulty processable sands having higher aluminum contents. The aluminum content of the tar sands is directly related to the fines content of the tar sands so that a measure of the sands aluminum content is proportional to the sands fines content and is therefore a measure of ease of processability. On the other hand it is thought that iron or zinc contents are merely incidentally variable with ease of processability—the iron or zinc content being generally low when sands aluminum content is high. A determination of the aluminum

content, however, is a more reliable indicator of processability because it is a direct measurement of the fines content. On the other hand, iron or zinc content in some sands can be high when fines content of the sands is high so that in this case the iron or zinc content are not indicative of the processability of the tar sands. Furthermore all clay minerals contain aluminum but they may or may not contain iron. In other words the iron and zinc contents of tar sands are not necessarily relatable to ease of processability.

The present invention can be described as an improvement to the hot water process wherein the hot water process comprises forming a mixture of water and tar sands, passing the mixture to a process separation zone to form an upper bitumen froth layer, a lower sand tailings layer and a middlings layer comprising water, mineral and bitumen and separately removing the layers from the separation zone. The improvement comprises determining the aluminum content of the tar sands and regulating the amount of water used to form the mixture of sands and water by increasing the amount of water with higher aluminum content and decreasing the amount of water with lower aluminum content so that optimum conditions are maintained for forming bitumen froth in the process separation zone. The determination of the aluminum content allows for an anticipatory control of the proper amount of water used to form the water and sands mixture.

In the hot water process, monovalent alkaline reagents are added to the tar sands conditioning drum usually in the amount of 0.1 to 3.0 pounds per ton of tar sand. The amount of such alkaline reagent preferably is regulated to maintain the pH of the middlings in the separator zone of the process within the range of 7.5 to 9.0. Best results seem to be obtained at a pH value of 8.0 to 8.5. The amount of the alkaline reagent that needs to be added to maintain a pH value in this range of 7.5 to 9.0 varies from time to time as the composition of the tar sands from the mine site varies. Thus as the fines content of the sands increases, reagent consumption is increased. The present invention can provide a convenient control of added reagent. By the present invention the tar sands aluminum content can be measured and clay content determined. The amount of added reagent is then determined and added in anticipation of pH change in the separation cell brought about by increased reagent consumption with tar sands of varying clay contents as determined by measured aluminum content. Thus a more delicate control can be maintained over the process resulting in a savings in reagent and the maintenance of a continuous optimum bitumen froth yield. Thus by the process of the present invention, water input into the hot water process, reagent addition and both water input and reagent addition can be controlled according to the determined processability of the tar sands feed.

Another embodiment of the present invention relates to the means used to determine the aluminum content of the tar sands. It has been found in the present invention that aluminum content of tar sands can be determined by rendering the tar sands or a portion of feed sands radioactive by neutron irradiation, recording emissions from the sands and analyzing the emissions to determine aluminum content and hence the clay content and ease of processability of the sands.

In one preferred embodiment, a neutron irradiation system, deuterium gas, is ionized and accelerated in a beam which is directed against a tritium target to produce fast neutrons. The neutrons are directed against the tar sands to cause them to become artificially radioactive. The gamma rays emitted by the radioactive sands have energies characteristic of the elements that emit them. Analyses of these emissions at various energy levels provide an energy spectrum characteristic of the elements present and proportional to the amount of the elements, thus permitting a quantitative analysis of the sands. Not only does this procedure allow determination of aluminum content but also silicon content, oxygen and carbon as well. The aluminum measurement provides a continuous measure of clay content and provides operators of hot water separation processes with an anticipatory indication of quantities of monovalent alkaline reagents and water needed to properly treat the bituminous tar sands to obtain maximum primary bitumen froth yield.

The invention will be described in more detail with reference to the drawing which shows a preferred embodiment of the present invention and schematically illustrates controlling the fresh water input to a hot water process for the separation of tar sands. In the drawing tar sands are charged into the process from the mining area via line **1**. The sands or a portion of the feed sands pass preferably on a conveyor belt through the tar sands analysis zone **2** where the aluminum content of the sands is determined and the water feeds of the process are varied according to this determination as will be described in greater detail infra. The tar sands analysis zone can comprise means useful for determining the aluminum content of the tar sands but preferably the zone consists of the neutron irradiation system as shown and described supra. With this type of system the conveyor carrying the tar sands moves the sands material past a leveling bar and under a vertically-mounted neutron generator. The neutron flux from this generator is incident upon the moving tar sands and causes fast neutron inelastic scattering from the carbon and oxygen atoms and activates other elements such as silicon and aluminum. The prompt gamma rays emitted by the carbon and oxygen atoms in the sands are measured with a prompt gamma ray spectrometer at the point of irradiation while the delayed activity due to silicon and aluminum is measured downstream before the sands are fed into the conditioning drum **4**. A data acquisition and analysis system is used consisting of an automated 100 kv. neutron generator, a neutron monitor, a prompt gamma ray spectrometer, a delayed gamma ray spectrometer, and a data analysis unit. The neutron generator is positioned with the target assembly approximately ½ inch above the plane of the sands. The nuclear analysis system is capable of measuring the yield of two elements each from the prompt and from the delayed gamma ray radiation from an irradiated material. The counting yields from each element measured are recorded by scalers which correct for background radiation and Compton gamma rays from higher energy gamma radiation. The neutron flux, which is held at a constant level through servo-controls in the generator control circuit, is also recorded on a scaler. At the end of a preselected counting interval the outputs of the five scalers, the 24-hour time, and the elapsed time are printed and the system reset for the next count by action of the data analysis control circuit.

The tar sands pass on the conveyor illustrated by line **3** from the analysis zone **2** to the tar sands conditioning drum **4**. Water and steam are introduced into the conditioning drum from **5** and mixed with the sands. Enough steam is introduced to raise the temperature in the conditioning drum to above 170° F. Monovalent alkaline reagent can also be added to the conditioning drum to maintain the pH of the middlings layer in separator zone **14** within the range of 7.5 to 9.0. Mulling of the tar sands produces a pulp which then passes from the conditioning drum as indicated by line **6** to a screen indicated at **7**. The purpose of screen **7** is to remove from the tar sand pulp any debris, rocks, or oversized lumps as indicated generally at **8**.

The pulp then passes from screen **7** as indicated by **9** to a sump **10** where it is diluted with additional water from **11** and a middlings recycle steam **12**. The pulped and diluted tar sands are pumped from the sump **10** through line **13** into the separation zone **14** which comprises a settling cell which contains a relatively quiescent body of hot water which allows for the formation of a bitumen froth which rises to the cell top and is withdrawn

via line 15 and a sand tailings layer which settles to the bottom to be withdrawn through line 16. An aqueous middlings layer between the froth and tailings layer contains silt and clay and some bitumen which failed to form froth. Since sufficient clay is not removed in the sand tailings withdrawn from the bottom of the separation cell through 16 in order to prevent the buildup of clay in the system it is necessary to continually remove some of the middlings layer and supply enough water in the conditioning operations to compensate for that so removed. The rate at which the middlings need to be removed from the system depends upon the content of clay and silt present in the tar sands feed and this rate varies as the content of these fines varies as determined per the present invention. If the clay and silt content is allowed to build up too high in the system effective separation will no longer occur and the process will become inoperative. This is avoided by regulating the recycling and withdrawal of middlings and input of fresh water per the present invention. Even when the separation step is operating properly the middlings layer withdrawn through line 17 will contain a substantial amount of bitumen which did not separate. Hence the middlings layer withdrawn through line 15 is for the purpose of description, herein referred to as "oil-rich" or "bitumen-rich" middlings.

The bitumen-rich middlings stream withdrawn from the separator 14 through line 17 is sent to a scavenger zone 16 wherein an air flotation operation is conducted to cause the formation of additional bitumen froth. The rate of flow of this stream from the separation zone is controlled by variable speed pump 19 which is in turn regulated by lead 20 which is responsive to the tar sands aluminum content of feed tar sands as determined in analysis zone 2. For example if the aluminum analysis of the feed tar sands indicates a feed of increased fines content, lead 20 increases variable speed pump 19 thereby increasing the flow in line 17 to the scavenger zone 18. Increased flow in the scavenger cell 18 lowers the interface level between the middlings and froth in separation zone 14. The lowering of the interface level actuates float valve 20 which by means of lead 21 opens valve 22 thus increasing the flow of fresh water addition to sump 10 via line 11. Increased water flow through 11 results in increased water content in the diluted pulp passing from the sump 10 through line 13 to the separation cell 14. Flow through valve 23 is decreased via lead 24 which responds to the increase in water in the diluted pulp thereby resulting in a reduction in the amount of middlings recycle diluting the separation cell feed via 12. The net effect of this is an increase in the proportion of fresh water in the separation cell 14 so that a constant middlings density is maintained with the introduction of the high clay containing tar sands feed which was analyzed in zone 2. Correspondingly if the analysis of aluminum conducted in zone 2 indicates tar sands feed of decreased clay content, lead 20 decreases the variable speed pump 19 thereby decreasing the flow in line 17 to scavenger cell 18. Decreased flow to the scavenger cell raises the interface level in the separation cell 14. A raising of the interface level actuates float valve 20 which by means of lead 21 closes valve 22 thus decreasing the flow of fresh water addition to the sump via line 11. Decreased water flow through line 11 results in decreased water content in the diluted pulp passing from the sump 10 through line 13 to the separation cell 14. Flow through valve 23 is increased via lead 24 which responds to the decrease in water in the diluted pulp in 13 thereby resulting in an increase in the amount of middlings recycle diluting the separation cell feed. Thus the proportion of fresh water in the separation cell 14 is decreased to accommodate the tar sands of decreased fines content so as to maintain a constant middlings viscosity in the separation cell. This viscosity can range between 0.4 to 5.7 centipoises, preferably 1 to 2 centipoises.

Following the process further, in the scavenger zone 18 an air flotation is conducted by any of the air flotation procedures conventionally utilized in processing of ores. The air causes the formation of additional bitumen froth which passes from the scavenger zone 18 through line 25 to a froth settler zone 26. A bitumen-lean middlings stream is removed and discarded from the bottom of the scavenger zone 18 via line 27.

In the settler zone 26 the scavenger froth forms into a lower layer of settler tailings which is withdrawn and recycled via line 28 to be mixed with bitumen-rich middlings for feed to the scavenger zone 18 via line 17. In the settler zone an upper layer of upgraded bitumen froth forms above the tailings and is withdrawn through line 29 and is mixed with primary froth in line 15. The combined froths are at a temperature of about 160° F. They are heated with steam and diluted with sufficient naphtha or other diluent from 30 to reduce the viscosity of the bitumen for centrifuging in zone 31 to produce a bitumen product 32 suitable for further processing.

What is claimed is:

1. In a hot water process for separating bitumen from bituminous tar sands which comprises forming a mixture of water and said tar sands; passing the mixture to a process separation zone to form an upper bitumen froth layer, a lower sand tailings layer and a middlings layer comprising water, mineral and bitumen and separately removing said layers from the said separation zone, the improvement which comprises:

(a) determining the aluminum content of said tar sands; and

(b) regulating the amount of water used to form said mixture by increasing said amount of water with higher aluminum content and decreasing said amount of water with lower aluminum content so that optimum conditions are maintained for forming bitumen froth in said separation zone.

2. The process of claim 1 in which step (a) of the improvement comprises rendering a sample of said tar sands radioactive by neutron irradiation; recording emissions from said radioactive sample; and analyzing said emissions to determine the aluminum content of said sample.

3. The process of claim 1 in which step (b) comprises regulating the amount of water used to form said mixture by increasing said amount of water with higher aluminum content and decreasing said amount of water with lower aluminum content to maintain the viscosity of said middlings layer in the range of about 0.4 to 5.7 centipoises.

4. The process of claim 1 in which step (b) comprises regulating the amount of water used to form said mixture by increasing said amount of water with higher aluminum content and decreasing said amount of water with lower aluminum content to maintain the viscosity of said middlings layer in the range of about 1 to 2 centipoises.

5. A method for determining the processability of bituminous tar sands which comprises rendering said tar sands radioactive by neutron irradiation, recording emissions from said radioactive sands; and analyzing said emissions to determine the aluminum content of said sands and hence the clay content and ease of processability of said sands.

6. The process of claim 1 in which a monovalent alkaline reagent is added to said mixture of water and tar sands to maintain the pH of said middlings layer within the range of 7.5 to 9.0.

7. The process of claim 6 which additionally comprises regulating the amount of monovalent alkaline reagent added by increasing said amount of reagent with higher aluminum content and decreasing said amount of reagent with lower aluminum content to maintain the pH of said middlings layer within the range of 7.5 to 9.0.

8. In a system for conducting a hot water process for treating tar sands comprising a conditioning drum, a separation cell, a first line for supplying tar sands pulp from said conditioning drum to said separation cell, a second line for introducing hot water into tar sands pulp in

said first line, a third line for withdrawing a bitumen froth product from said cell, a fourth line for withdrawing a sand tailings layer from said cell, a fifth line for withdrawing a middlings portion from said cell, a sixth line for recycling a middlings portion from said cell to be mixed with said tar sand pulp prior to discharge into said cell; the improvement which comprises:

(a) a tar sands analysis means for determining feed tar sands aluminum content and hence said sands clay content and

(b) regulating means controllably attached to said fifth line and responsively connected to said analysis means to increase the flow of middlings withdrawn via said fifth line when said feed tar sands are analyzed to have higher clay contents and to decrease the flow of middlings withdrawn via said fifth line when said feed tar sands are analyzed to have lower clay contents;

(c) regulating means operating in response to said middlings withdrawn in said fifth line and connected to said second line to control the hot water introduced to the bituminous tar sands pulp via said second line; and

(d) regulating means operating in response to said hot water incorporated in said second line and connected to said sixth line to control the middlings portion recycled to the bituminous tar sands pulp via said second line.

9. The system of claim **8** in which said tar sands analysis means (a) comprises a conveyor for moving tar sands through an analysis zone, a source of fast neutrons positioned to irradiate tar sands on said conveyor to artificially radio-activate said tar sands, a radiation measuring device positioned so as to measure energy emissions from said radio-active tar sands, and a recording device for recording said measured emissions for analysis for determining aluminum content of said tar sands according to recorded emissions characteristic of said aluminum.

10. In a hot water process for treating bituminous tar sands which comprises forming a pulp of feed bituminous tar sands with a minor amount of water in a pulping zone, removing pulp therefrom and mixing the same with hot water and a hereinafter specified recycle stream in a dilution zone, passing the mixture into a separation zone, settling the mixture in the separation zone to form an upper bitumen layer, a middlings layer comprising water, clay and bitumen, and a sand tailings layer, removing a first stream of middlings layer from the separation zone and passing it to the dilution zone as the aforesaid recycle stream, passing a second stream of middlings layer to a scavenger zone and therein recovering an additional amount of bitumen froth, and regulating the rate of passage of said second stream to the scavenger zone so as to regulate and maintain the viscosity of said middlings layer within the range of about 0.4 to 5.7 centipoises, the improvement to the regulating step which comprises: measuring the aluminum content of feed bituminous tar sands and determining sand clay content thereby; and regulating the rate of passage of said second stream to said scavenger zone according to said measured aluminum content.

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