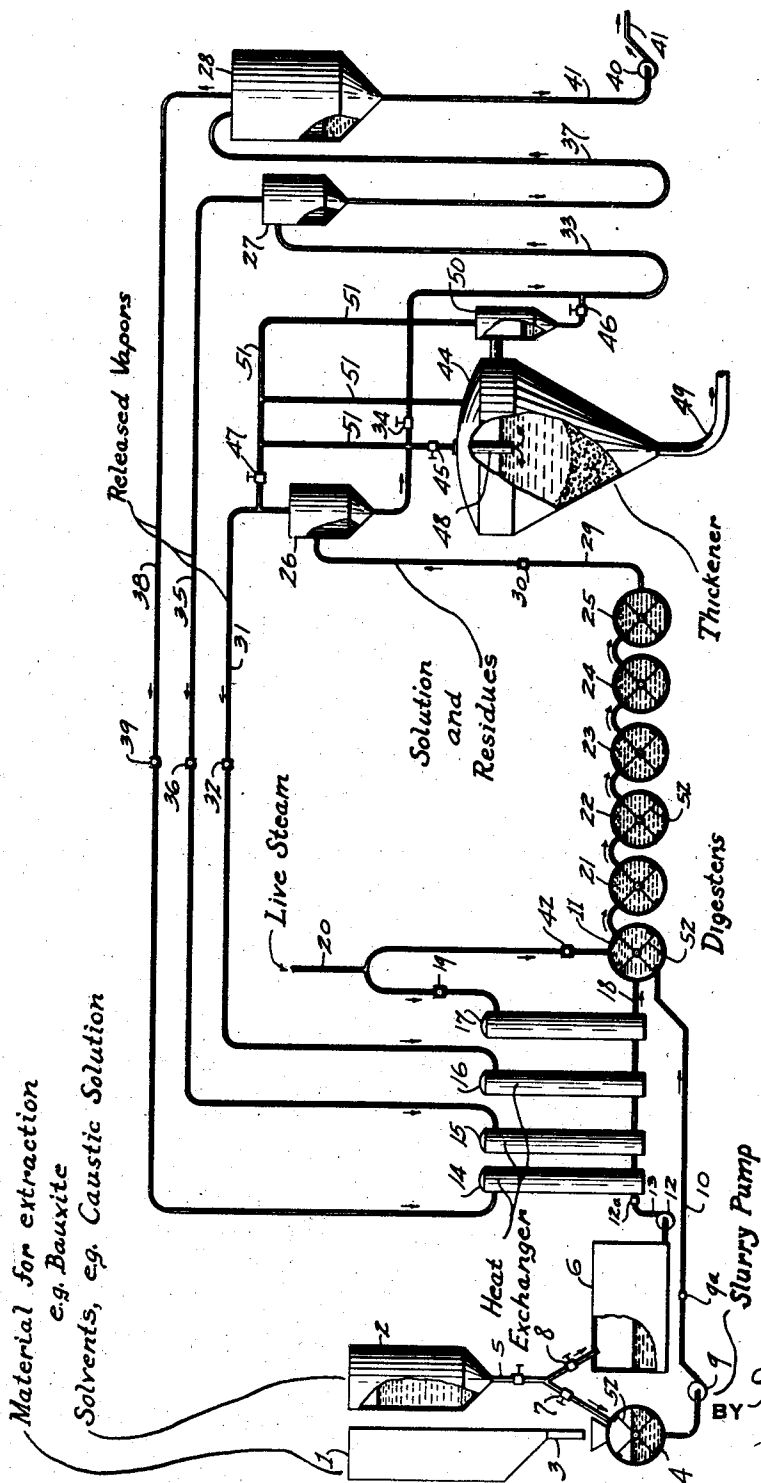


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CONTINUOUS DIGESTION PROCESS

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CONTINUOUS DIGESTION PROCESS

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This invention relates to the extraction of a solid with a solvent in a process of continuous pressure digestion. It relates particularly to, and has as its chief object, the provision of an improved method of continuously extracting the alumina from alumina-bearing materials by pressure digestion with caustic liquor, and an improved system adapted to carry out such a process.

The copending application of Weigel, Serial No. 561,081, discloses a continuous method of digesting bauxite with caustic liquor. This continuous process consists broadly in heating the mixture of bauxite and caustic liquor in successive stages of increasing temperature and pressure until the desired digestion temperature and pressure are attained, holding until the digestion is complete, and then cooling in successive stages of decreasing temperature and pressure. In this process, steam released in the cooling stages is injected in some of the preliminary heating stages, thus materially increasing the utilization of the heat supplied to the system. Difference in pressure throughout the system is secured as a rule hydrostatically by positioning the component parts of the system in a plurality of different elevations. This invention allowed materially increased thermal and material efficiencies to be secured and was a great advance over the older batch system.

The present invention is similar to the above-described process of Weigel in that it is capable of continuous operation through holding and cooling stages and in that heat released from the cooling stages may be utilized in the digestion. Our invention contemplates an essentially simplified process with provision for carrying it out in a system constructed substantially on one level with few moving parts and requiring a minimum of capital expense and operating labor. Provision is made for efficiently heating the digestion with steam without direct contact so that dilution due to the condensation of the steam is avoided. Owing to the elimination of the direct injection of steam as a heating means it has been found possible to usefully employ very low pressure steam for heating, as there is no direct mechanical relationship between the pressure in the steam system and the pressure in the digesting system. This gives a greater flexibility of control than would otherwise be possible. With independent control over the temperatures and pressures and working with a substantially uniform flow of undiluted digest mixture it has been found practical to make a separation of the caustic digest liquor

from the solid residue after digestion at an elevated temperature equal to or greater than the normal boiling point of the liquor under atmospheric pressure, as will be further described hereinafter.

It has long been a desideratum to apply steam heat indirectly to a batch pressure digestion to avoid the dilution arising from heating by direct injection of steam. This has proved difficult, as the digestion mixture precipitates solids on the heated surfaces, which seriously interfere with the transfer of heat. In a batch process this difficulty may be overcome to a certain extent by employing high pressure steam and carefully cleaning the heating surfaces between batches. In a continuous heat regenerative digestion process this problem has presented a serious difficulty because no opportunity is afforded for cleaning the heating surfaces and a layer of solid material of increasing thickness is continually formed. Furthermore, in a heat regenerative system the difference in temperature between the material to be heated and the heating medium is small, and a material resistance to heat flow or transfer between the two substantially negates the advantages of such a regenerative process. In our solution of this problem it was found that the caustic liquor, which at times contains small amounts of alumina and other substances in solution from prior treatments, may be satisfactorily superheated in respect to the desired digestion temperatures in known types of heaters, such as a straight tube heat exchanger, without the formation of scale on the heater walls. It was also found that a substantially complete extraction of all available alumina from alumina-bearing material might be made by treating such material with superheated caustic liquor to produce a digestion mixture at a predetermined digestion temperature. In actual practice it has been found desirable to wet the alumina-bearing material with sufficient caustic liquor to form a thick slurry which can be conveniently introduced into the digestion process under pressure, as by pumping.

In the preferred practice of our invention the caustic liquor is divided. This liquor may be at room temperature or at a somewhat elevated temperature, depending upon the general operation of which this process may form a part. One portion is passed under pressure through a step of indirect heating, which may consist of several stages of increasing temperature until the liquor has been given a superheat with respect to the contemplated digestion temperature. The other

portion, preferably the smaller portion, is added to the alumina-bearing material to form a thick slurry which is subsequently maintained under pressure. The two portions are then combined under pressure and the superheat of the liquor portion is absorbed by the slurry portion to produce a digestion mixture having a temperature predetermined by the degree of superheat in the liquor. The digestion mixture is passed through a plurality of holding stages under these conditions of temperature and pressure until substantially all of the available alumina is dissolved by the caustic liquor. Under these conditions the digest solution is rendered stable and there is no precipitation of alumina, which assures a maximum yield. The mixture is then passed through a plurality of cooling stages. Throughout the entire cycle of operation a constant flow of digestion mixture from step to step and from stage to stage is preferably maintained. During each cooling stage heat is absorbed in the evaporation of water from the liquor under reduced pressure. This heat may be applied to the liquor-heating stages and, owing to the use of indirect heating, steam generated at substantially any pressure in the cooling stages may be efficiently utilized in supplying heat to the heating steps. Even very low pressure exhaust steam may be used in the initial stages of liquor heating.

For the purpose of explanation, reference will be had to the drawing which forms a part of this specification and which represents, in schematic outline, a flow sheet of a process embodying this invention in the extraction of alumina from alumina-bearing material.

Referring to the drawing, the apparatus which may be used in the practice of our invention comprises proportioning feed hopper 1 for the alumina-bearing material and caustic liquor proportioning tank 2, slurry mixer 4, surge tank 6, tubular liquor heaters 14, 15, 16 and 17, digesters 11, 21, 22, 23, 24 and 25, cooling chambers 26, 27 and 28, thickener 44, and tank-connecting and mixture-transferring means hereinafter referred to. The slurry mixer and the digesters are equipped with revolving stirring arms 52 to prevent the solids settling from the liquor. It will be understood that while the process will be described as having specifically four heating stages and three cooling stages and six digesting vessels, this is merely by way of example, and fewer or more stages may be used to best satisfy any particular condition.

The reagents, that is, alumina-bearing material, which may be bauxite, and caustic liquor, by which is meant solutions of potassium or sodium hydroxide which may contain in addition small amounts of other substances, such as carbonates and aluminates, are drawn continuously at a constant rate or in batches from the tanks 1 and 2 respectively. The bauxite passes through the proportioning apparatus 3 directly into a slurry mixer 4. The caustic liquor passes through the pipe line 5 which has two branches, one leading to the slurry mixer 4 and the other to the clear liquor surge tank 6. The two branch lines are equipped with proportioning orifices 7 and 8 which permit a measured flow of both streams. Orifice 7 preferably discharges about one-tenth of the liquor and orifice 8 about nine-tenths. A pump 9 draws the slurry from the mixer 4 and forces it through the pipe line 10 into a pressure digesting vessel 11. Pump 12 forces clear caustic liquor from the surge tank 6 through the line 13 to the heater

14 which may be a tubular heater, as indicated, through which it passes and is partially brought to a superheated condition by heat supplied by low pressure steam through the tube walls. In practice it is desirable to use oversize pumps and reduce the flow by valves in the pump discharge line, such as 9a and 12a. From the heater 14 the liquor passes through similar heaters 15 and 16 which are heated progressively by somewhat higher pressures of steam to higher temperatures, after which the liquor finally passes through a heater 17 which is heated with still higher pressure steam, which raises the temperature of the liquor to a superheat with respect to the other temperatures employed in the process. The superheated liquor from 17 passes through pipe line 18 into the digester 11. The superheat of the liquor is given up to the slurry from mixer 4 and may be adjusted to give the desired digestion temperature by means of the degree of superheat, which in turn is regulated by the thermostatic steam valve 19 on the steam supply line 20.

From the digestion vessel 11 the mixture passes through similar digesters 21, 22, 23, 24 and 25 under substantially the conditions of temperature and pressure maintained in digester 11. During this passage the alumina is in a large part extracted in digesters 11 and 21. Digesters 21, 22, 23, 24 and 25 serve as holding digesters to allow sufficient time to elapse to substantially complete the digestion or solution of all of the available alumina and to stabilize the digestion mixture.

The digestion mixture, which now consists of a caustic aluminate solution and the insoluble residue from the bauxite, passes from digester 25 to the cooling tanks or blow-off tanks 26, 27 and 28. The digestion mixture is released continually from digester 25 through the pipe line 29 and the control or blow-off valve 30 to tank 26. The pressure in tank 26 is lower than the digestion pressure and steam is released. The steam passes through pipe 31 and control valve 32 to the tubular heater 16. The pressure in tank 26 is largely controlled or set by valve 32 and is higher than the pressure in tank 27. The mixture therefore flows from tank 26 to tank 27 through the loop pipe seal 33 and the valve 34. Steam is again released at lower pressures and passes through the pipe line 35 and valve 36 to heater 15. The cooling mixture is still under substantial pressure and flows through loop seal pipe line 37 to tank 28. Steam is released in this tank and is led to heater 14 through the pipe 38 and the control valve 39. The mixture is drawn from tank 28 by a pump 40 through pipe 41 and delivered to filters for separating the residue from the liquor, not shown, which is a step preliminary to treating the caustic liquor for the recovery of alumina therefrom in known manner, which treatment forms no part of this invention.

In this process the mixture of bauxite and liquor is digested without dilution from the condensate of heating steam and, in fact, a certain not unsubstantial amount of concentration is effected by the amount of steam released in the blow-off tanks. It is sometimes desirable, for instance, in order to reduce capital expense, to heat the mixture of caustic liquor from the regenerative heaters 14, 15 and 16 and slurry from mixer 4 by the direct injection of high pressure steam in the digester 11 and thereby eliminate the superheater 17. In this case the digestion

temperature is controlled by the thermostatic valve 42 in the steam supply line 20. In this modification of our invention all of the advantages of the various steps of heat regeneration are preserved unaffected, and because of the fact that the digestion mixture is partially heated indirectly and to a degree concentrated, satisfactory operation may be secured by the injection of high pressure steam to effect the last stage of heating. If desired, a combination of both means of heating may be used.

Primarily, the flow of materials through the process is controlled by the pumps 9 and 12 and valves 9a and 12a, which maintain a pressure at least equal to the pressure in the digesters, and the blow-off valve 30. Secondly, the flow is controlled by the valves 32, 36 and 39 and the pump 40. If desired, the elevation of the blow-off tanks 26, 27 and 28 may be adjusted to secure the desired action through the loop seals 33 and 37. The temperature of the process is controlled primarily by valves 19 and 42 which regulate the primary source of heat. Secondly, the temperature is controlled through the valves 32, 36 and 39 and the orifices 7 and 8. The flow of materials is continuous throughout the process. The digesters are kept filled with material at all times by the pumps 9 and 12 and the blow-off valve 30. The range of possible adjustments of valves 32, 36 and 39 is very wide, as the steam entering the heaters 14, 15 and 16 has no mechanical relation to the pressure maintained on the digestion solution, and the heat can therefore be supplied by steam at any desired pressure having the desired temperature within the range of the pressure of the released steam. This gives the utmost flexibility in controlling the conditions of material flow, temperature and pressure.

In such a process having a continuous flow of materials at predetermined controllable pressures and temperatures as described, it has been found practical to insert a step of thickening or separation of the solid residue dispersed in the caustic aluminate liquor after the digestion is complete. The residue may be, therefore, in this case, discharged separately from the process as a thick sludge containing a small amount of liquor and the extract liquor discharged substantially clear and containing very little residue. The technical prerequisites for such a separation are that the flow of material through the thickener be constant, or nearly so, and that the temperature and the concentration of dissolved material be such that no precipitation occurs in the thickener. The practical prerequisite is that in addition the amount (i. e., the volume) to be treated in the thickener be small, and that the settling rate be rapid; otherwise, the size of thickener necessary may become larger than can be economically operated under the conditions of temperature and pressure, or the time required may be so long as to negate any advantage in its use. In the case of extracting alumina with caustic liquor as described, the thickening step may be placed, for example, between the first two stages of cooling, that is, between blow-off tanks 26 and 27. This is shown in the drawing. The enclosed thickener 44 may be connected in the system in the loop seal pipe line 33 by closing valve 34 and opening valves 45, 46 and 47. This mixture discharged from tank 26 flows through valve 45 and pipe 48 into the thickener below the liquid level where the residue settles in the cone bottom and is drawn off as a sludge

through the discharge pipe 49 by any suitable form of sludge pump, not shown. The clear liquor overflows into the tank 50 and flows through the valve 46 into the loop seal pipe line 33 to tank 27. The pipes 51 equalize the pressure throughout the thickening steps and maintain a pressure substantially equal to the pressure of tank 26, and the thickener is of course thus maintained under approximately the temperature of tank 26. The conditions of thickening above set forth have been found to materially increase the settling rate of the solid material from the mixture, which serves to decrease the size of the thickener or settling equipment. For example, the settling rate may be increased fifty per cent or more by elevating the temperature. The temperature is preferably increased above the normal boiling point of the liquid at atmospheric pressure. This remarkable change in settling rate is thought to be due principally to a change in viscosity of the liquor with temperature, but it is not desired to limit the fact in any way by this explanation.

It will be noted that in the operation of this process now being described it is necessary only to apply heat at one point, heater 17 or digester 11, which is done with steam pressure as from the steam supply line 20, which in turn may be connected to any source of steam of sufficient pressure.

Although we have described our invention with reference to a particular embodiment thereof, it will be apparent that it is by no means limited to the details of operation, temperature, pressure, composition or substances described but may be utilized to advantage in any and all similar methods of pressure digesting aluminous material or other solids with a liquid.

We claim:

1. A method of continuously pressure digesting solids with a solvent, comprising forming a slurry of the solids with some of the solvent, and indirectly heating another portion of the solvent in a plurality of successive stages of increasing temperature to a predetermined superheat with respect to the slurry, combining the slurry and the solvent under pressure to obtain a predetermined digestion temperature, digesting the mixture in a plurality of stages, then cooling the mixture in a plurality of separate stages of decreasing temperature and pressure, controlling the decrease in pressure between successive cooling stages so as to release vapor from the solvent in such quantity that it is adapted to supply heat to several of the solvent-heating stages, the heating of the solvent in several such stages being accomplished by passing said released vapor into heat exchangers which prevent contact between said vapor and said solvent, and maintaining a flow of materials throughout all the steps of the process.

2. A method of continuously extracting alumina from aluminous material, which comprises forming a slurry of said aluminous material and caustic liquor, indirectly heating a further amount of caustic liquor in a plurality of successive separate stages of increasing temperatures to a temperature such that when combined with the slurry a predetermined temperature of the mixture is attained, combining the slurry and the heated caustic liquor under pressure, and digesting under suitable conditions of temperature and pressure until substantially all of the alumina is in solution, then cooling the mixture in a plurality of separate stages of de-

creasing temperature and pressure, predetermining the decrease in pressure between successive cooling stages so as to release steam from the caustic liquor in such quantity that it is adapted to supply heat for several of the caustic liquor heating stages by passing said released steam into heat exchangers which prevent contact between said steam and said caustic liquor, and maintaining a flow of materials throughout all the steps of the process.

3. A method of continuously extracting alumina from aluminous material, which comprises forming a slurry of said aluminous material and caustic liquor, indirectly heating a further amount of caustic liquor in a plurality of successive separate stages of increasing temperature to a sufficient temperature so that when combined with the slurry a predetermined temperature of the mixture is attained, combining the slurry and the heated caustic liquor and digesting under suitable conditions of temperature and pressure until substantially all of the alumina is in solution, then cooling the mixture in a plurality of separate stages of decreasing temperature and pressure, the decrease in pressure between successive cooling stages being such that steam is released from the caustic liquor in such quantity that it is adapted to supply heat for several of the caustic liquor heating stages, the heating of the caustic liquor in several such stages being accomplished by passing said released steam and caustic liquor into heat exchangers which prevent contact between said steam and said caustic liquor, separating by sedimentation the undissolved portion from the liquor solution at a temperature higher than the boiling point of said liquor at atmospheric pressure, and maintaining a flow of materials throughout all the steps of the process.

4. Apparatus for extracting alumina from aluminous material, which comprises, in combination, means for forming a slurry of aluminous material and caustic liquor, means for indirectly heating caustic liquor in a plurality of separate stages of increasing temperature, means for combining and digesting the slurry and caustic liquor under controlled conditions of temperature and pressure, means for cooling the digestion product in a plurality of successive stages of decreasing temperatures and pressures, means for indirect heat exchange between said heating and cooling means, and means for maintaining a constant flow of material throughout.

5. Apparatus for extracting alumina from aluminous material, which comprises, in combination, means for forming a slurry of aluminous material and caustic liquor, means for indirectly heating caustic liquor in a plurality of separate stages of increasing temperature, means for controllably combining the slurry and caustic liquor and for digesting the mixture under controlled conditions of temperature and pressure, means for cooling the digestion product in at least two stages of decreasing temperatures and pressures, means for indirect heat exchange between said heating and cooling means, means for separating insoluble portions by sedimentation from the digested liquor solution, and means for maintaining a constant flow of material throughout.

6. A method of continuously extracting alumina from aluminous materials with caustic liquor, which comprises dividing the caustic liquor into major and minor portions, forming a slurry of the aluminous material with the minor portion and heating the major portion indirectly

to a temperature higher than that of the slurry such that when combined with said slurry the temperature of the resultant mixture is increased to a predetermined digestion temperature, combining said slurry and heated caustic liquor under pressure, digesting the mixture under controlled conditions of temperature and pressure until substantially all of the alumina is in solution, and maintaining a flow of materials throughout all the steps of the process.

7. In apparatus for extracting solids with solvents by continuous pressure digestion, the combination comprising a mixing unit adapted to form a pumpable slurry from a solid to be extracted and a solvent, a plurality of digesting tanks, means for continuously supplying said slurry to said digesting tanks, means for continuously supplying additional solvent to said digesting tanks in an amount having a predetermined ratio to the amount supplied to said mixing unit, a plurality of heat exchange units adapted to raise the temperature of said additional solvent in progressive steps, a plurality of cooling chambers connected in series, transfer means adapted to deliver solution and undissolved solids from said digesting tanks to said cooling chambers, means for regulating the pressure in the digesting tanks, control means between adjacent cooling chambers in said series adapted to maintain progressively decreasing pressures in said chambers, and means for conveying the vapor released in at least one of said cooling chambers to at least one of said heat exchange units.

8. In apparatus for extracting solids with solvents by continuous pressure digestion, the combination comprising a surge tank for solvent, a mixing unit adapted to form a pumpable slurry from a solid to be extracted and a solvent, means for supplying said solid to said mixing unit, means for supplying said solvent to said mixing unit, means for supplying solvent to said surge tank, a plurality of digesting tanks connected in series, means for continuously supplying said slurry to the first of said series of digesting tanks from said mixing unit, means for continuously supplying additional solvent to the first of said series of digesting tanks from said surge tank in an amount having a predetermined ratio to the amount supplied to said mixing unit, a plurality of heat exchange units interposed in the supply means for additional solvent and adapted to raise the temperature of said additional solvent in progressive steps, a plurality of cooling chambers connected in series, transfer means adapted to deliver solution and undissolved solids from the last of said series of digesting tanks to the first of said series of cooling chambers and including means for regulating pressure in the digesting tanks, control means between adjacent cooling chambers in said series adapted to maintain pressures in said chambers decreasing progressively in steps from the digestion pressure, and means for conveying vapor released by the decrease of pressure in at least one of said cooling chambers to at least one of said heat exchange units.

9. In apparatus for extracting solids with solvents by continuous pressure digestion, the combination comprising a mixing unit adapted to form a pumpable slurry from a solid to be extracted and a solvent, a plurality of digesting tanks, means for continuously supplying slurry to said digesting tanks from said mixing unit, means for continuously supplying additional solvent to said digesting tanks, a plurality of cooling chambers connected in series, transfer means

adapted to deliver solution and undissolved solids from said digesting tanks to the first of said series of cooling chambers, means for regulating the pressure in the digesting tanks, control means between adjacent cooling chambers in said series adapted to maintain pressures in said chambers decreasing in steps from the digestion pressure, and a closed sedimentation tank positioned between and operatively connected with two adjacent cooling chambers in said series and adapted to separate solids from solution under substantially the conditions of superatmospheric pressure and elevated temperature maintained in the first of said connected cooling chambers.

10. In apparatus for extracting solids with a solvent by continuous pressure digestion, the combination comprising a mixing tank adapted to form a slurry of solid and solvent, at least one heat exchange unit, at least one digesting unit, at least one cooling chamber, controlled means for delivering slurry from said mixing tank to said digesting unit, means for passing a controlled quantity of additional solvent through said heat exchange unit and into said digesting unit at a controlled temperature and pressure, and means for delivering material from said digesting unit to said cooling chamber while maintaining the desired pressure in the digesting unit.

11. The method of extracting alumina from aluminous material with caustic liquor, comprising mixing part of the caustic liquor with aluminous material to form a slurry, heating a further quantity of the caustic liquor, mixing the heated liquor with the slurry, digesting the mixture at an elevated temperature and superatmospheric pressure, and separating the resulting solution from undissolved residues by sedimentation at a temperature above the boiling point of the solution at atmospheric pressure.

12. The method of extracting alumina from aluminous material with caustic liquor, comprising mixing solid aluminous material with caustic liquor to form a slurry, heating a further quan-

tity of caustic liquor by indirect contact with steam, digesting said slurry and heated caustic liquor in a closed vessel maintained at the desired superatmospheric pressure and elevated temperature by direct contact with steam to dissolve alumina, reducing the pressure of the resulting solution and withdrawing steam liberated thereby, and returning the steam to indirectly heat a further quantity of caustic liquor.

13. The method of extracting alumina from aluminous material, comprising digesting aluminous material with preheated caustic liquor in a closed vessel, maintaining the pressure above atmospheric and the temperature above the boiling point at atmospheric pressure by injecting steam into said vessel, separating the resulting solution from undissolved residues by sedimentation at elevated temperature and superatmospheric pressure, reducing the pressure on said solution by withdrawing vapor liberated thereby, and employing said vapor to preheat a further quantity of said caustic liquor.

14. The method of extracting alumina from aluminous material, comprising digesting aluminous material with caustic liquor at superatmospheric pressure and at a temperature higher than the boiling point of the liquor at atmospheric pressure, and separating the resulting solution from undissolved residues by sedimentation at elevated temperature and superatmospheric pressure.

15. The method of extracting alumina from aluminous material, comprising digesting aluminous material with caustic liquor at superatmospheric pressure and at a temperature higher than the boiling point of the liquor at atmospheric pressure, and maintaining the resulting solution and undissolved residues at superatmospheric pressure and elevated temperature until said residues are separated from the solution by sedimentation.

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