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(54) Title: METHOD AND ARRANGEMENT OF RECOVERING RARE EARTH ELEMENTS FROM ION ADSORPTION CLAYS

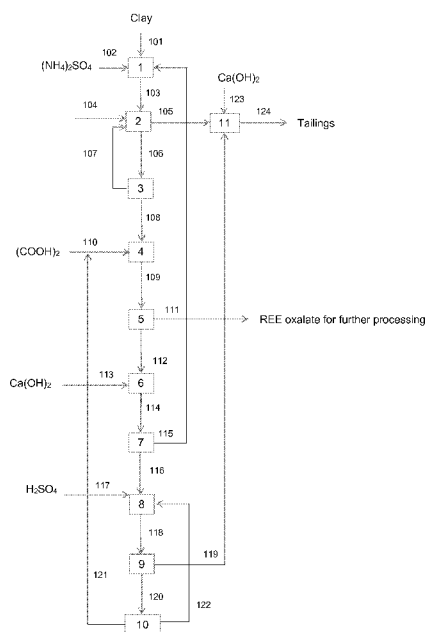


Fig. 1

(57) Abstract: A method and arrangement of recovering REEs wherein the ion adsorption clay is leached in a leach solution for producing a leach slurry, which leach residue contained in the leach slurry is washed with water and a pregnant leach solution and the leach residue are separated from each other. Water is separated from the pregnant leach solution and a concentrated pregnant leach solution containing the dissolved solids is obtained. Rare earth elements are precipitated as rare earth oxalate(s) and they are separated from the rare earth element depleted leach solution. The rare earth element depleted leach solution obtained from the rare earth element recovery step is contacted with calcium compound for precipitating unused excess oxalic acid as calcium oxalate which is separated from the remaining leach solution. The separated calcium oxalate is contacted with sulphuric acid for producing oxalic acid, which may be further crystallized and precipitated calcium sulphate.

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METHOD AND ARRANGEMENT OF RECOVERING RARE EARTH ELEMENTS FROM ION ADSORPTION CLAYS

FIELD OF THE INVENTION

The present invention relates to a method and arrangement of recovering rare earth elements from ion adsorption clays.

BACKGROUND OF THE INVENTION

Rare earth elements (REE) occurring in ion adsorption clays are typically extracted by simple leach processes with monovalent sulphate and/or chloride salt solutions at ambient temperature via ion exchange reaction. The leaching process can be either a reactor, in situ or heap leaching process. Predominantly rare earth elements have been recovered from ion adsorption clays by in-situ processes. These however are typically not environmentally friendly processes and the recovery of rare earth elements from ion adsorption clays has proven to be rather difficult. In-situ processes typically result in polluted ground water in the area, as well as erosion of the ground causing landslips etc. Heap processes on the other hand are prone to weather changes. A further challenge relating to the recovery of rare earth elements from ion adsorption clays is that the clay contains very low concentrations of rare earth elements. This causes challenges with respect to the controlling of water balance of the process as the material streams in the process are very large. A further challenge related to the recovery of rare earth elements from ion adsorption clays is that the leach slurry formed contains the solids in very fine particle size. This sets high demands for the needed solid-liquid separation processes. For example filtration and washing of such a material is extremely slow if not completely impossible.

Typically the refining of rare earth elements requires chemicals such as ammonium bicarbonate and oxalic acid.

BRIEF DESCRIPTION OF THE INVENTION

An object of the present invention is thus to provide a method and an apparatus for implementing the method so as to alleviate the above disadvantages and enable the recovery of rare earth elements from ion adsorption clays with high yield, economically and in environmentally friendly manner. The objects of the invention are achieved by a method and an arrangement, which are characterized by what is stated in the independent claims. The preferred embodiments of the invention are disclosed in the dependent claims.

The invention is based on the idea of recovering rare earth elements by the method and arrangement wherein the ion adsorption clay is leached in a leach solution for producing a leach slurry, the leach slurry is subjected to a solid-liquid separation step for washing leach residue contained in the leach slurry with water and for separating a pregnant leach solution and the leach residue from each other, water is separated from the pregnant leach solution thereby producing a concentrated pregnant leach solution containing the dissolved solids, the concentrated pregnant leach solution is contacted with oxalic acid for precipitating rare earth element(s) as rare earth oxalate(s) and the precipitated rare earth oxalate(s) are separated from the rare earth element depleted leach solution, the rare earth element depleted leach solution obtained from the rare earth element recovery step is contacted with calcium compound, such as calcium hydroxide, calcium carbonate or calcium chloride, for precipitating unused excess oxalic acid as calcium oxalate and the calcium oxalate is separated from the remaining leach solution, the separated calcium oxalate is contacted with sulphuric acid for producing oxalic acid, which may be further crystallized, and precipitated calcium sulphate.

An advantage of the method and arrangement of the invention is that rare earth elements are recovered with great yield in an economic and sustainable manner and in the method as little as possible make-up chemicals and water are needed. A further advantage is that reverse osmosis quality water is released from the process together with the solids, such as gypsum and leach residue solids. The solids are released from the process in such a form that they cause no harm to the surrounding environment.

25 BRIEF DESCRIPTION OF THE DRAWING

In the following the invention will be described in greater detail by means of an example embodiment with reference to the attached drawing, wherein Figure 1 is a flow diagram of an example embodiment of method of the invention.

30 DETAILED DESCRIPTION OF THE INVENTION

The present invention relates to a method of recovering rare earth element(s) from ion adsorption clay comprising

- a leaching step, wherein the ion adsorption clay is leached in a leach solution for producing a leach slurry,

- a solid-liquid separation step, wherein the leach slurry is subjected to a solid-liquid separation step for washing leach residue contained in the leach slurry with water and for separating a pregnant leach solution and the leach residue from each other,

5 - a concentration step, wherein water is separated from the pregnant leach solution thereby producing a concentrated pregnant leach solution containing dissolved solids,

 - a rare earth element recovery step, wherein the concentrated pregnant leach solution is contacted with oxalic acid for precipitating rare earth
10 element(s) as rare earth oxalate(s) and the precipitated rare earth oxalate(s) are separated from a rare earth element depleted leach solution,

 - oxalate recovery step, wherein the rare earth element depleted leach solution obtained from the rare earth element recovery step is contacted with calcium compound for precipitating unused excess oxalic acid as calcium
15 oxalate and the calcium oxalate is separated from a remaining leach solution,

 - oxalic acid regeneration step, wherein the separated calcium oxalate is contacted with sulphuric acid for producing oxalic acid and precipitated calcium sulphate.

20 The present method is typically performed under atmospheric pressure and in ambient temperature.

 Typically the ion adsorption clay comprises Al-silicate minerals e.g. kaolinite, K-silicate minerals e.g. microcline, Al-minerals e.g. gibbsite or any combination thereof having REE adsorbed on it. The ion adsorption clay may
25 comprise any of the above mentioned either alone or in any combination thereof.

 Typically the leaching step of the present method is performed as reactor leaching. In this manner a better contact between the ion adsorption clay and the leach solution is achieved, compared to in-situ or heap leaching. In reactor leaching rare earth elements are recovered with better yield compared to in-situ or heap leaching steps, wherein leach solution and dissolved
30 REEs with it are lost to some extent.

 It is possible to use any suitable leaching solution in the leaching step. Suitable leaching solutions contain 1+-charged ions, such as ammonium ions, sodium ions, potassium ions, or mixtures thereof. Typically, in the leaching
35 step the leach solution is sulphate solution, mixture of sulphate solutions,

chloride solution, mixture of chloride solutions or a mixture of sulphate and chloride solution(s). Examples of sulphate solutions are ammonium sulphate solution, sodium sulphate solution or a mixture thereof, etc. Examples of chloride solutions are potassium chloride, sodium chloride etc. With these solutions
5 excellent leaching yields were achieved and the risk of forming poorly soluble compounds in the leaching is very low. Typically the leach solution is 0.2 to 1 M solution of the active leaching agent. In the leaching step the positively charged ion of the leaching solution is carried into the clay and the rare earth elements are carried from the clay into the leach solution, i.e. via ion exchange
10 reaction. As the ion adsorption clay contains very low amounts of rare earth elements, the leach solution does not dilute significantly during the leaching step, in other words it remains seemingly the same throughout the leaching step.

In the leaching step a leach slurry is formed, which contains leach
15 solution, such as ammonium sulphate solution, containing solids, such as rare earth elements, in dissolved form and undissolved solid matter.

The method further comprises a solid-liquid separation step, wherein the leach slurry is subjected to a solid-liquid separation step for washing leach residue contained in the leach slurry with water and for separating a
20 pregnant leach solution containing solids, such as rare earth elements in dissolved form from the leach residue, i.e. the undissolved ion adsorption clay. The undissolved solid matter is removed from the process as leach residue. The ion adsorption clay lowers the pH of the leach solution typically to a pH value of approximately 5. Therefore the separated leach residue is typically
25 directed to a neutralization step before directing it further to tailings.

The leach slurry formed contains solid matter in a very fine particle size. This renders the solid-liquid separation very demanding as it is almost impossible to filter and wash the leach slurry. The leach solution used has to be washed away from the slurry, in other words all the rare earth element –
30 containing solution has to be replaced with rare earth element free solution, i.e. water. Thus, a huge amount of water is needed and filtering of such a huge amount is very expensive. Therefore in the method of the present invention the solid-liquid separation step is typically done in a circuit of counter-current decantation thickeners. Typically 1.5 to 2.5 m³ wash water is used per ton of solids. Thereby the obtained washing efficiency with respect to the amount of
35 wash water used is in high level and better than when filtration would be used. The dissolved rare earth elements are recovered with excellent yield and the

remaining solid matter is washed to be able to landfill in an environmentally sustainable manner. Typically the circuit of counter-current decantation thickeners contains 2 to 10 devices, more typically 5 to 6.

The water used for washing in the solid-liquid separation step is recovered in the concentration step of the method. This enables effective, economic and environmental friendly controlling of the water balance of the entire method. The concentration step is also important for enabling the use of equipment with smaller size in the subsequent process steps. Thus, after the solid-liquid separation step the method comprises a concentration step, wherein water is separated from the pregnant leach solution thereby producing a concentrated pregnant leach solution containing dissolved solids, such as rare earth elements. Typically the concentration step is performed by evaporation or with membrane filtration technology. More typically the concentration step is performed with reverse osmosis. With reverse osmosis as high as 95 to 99 weight-% of the total dissolved solids can be recovered. In this way pure water can be obtained from the process and recycled to the previous solid-liquid separation step to be used for washing.

The method comprises a rare earth element recovery step, wherein the concentrated pregnant leach solution is contacted with oxalic acid for precipitating rare earth element(s) as rare earth oxalate(s). After that the precipitated rare earth oxalate(s) is/are separated from the rare earth element depleted leach solution. The rare earth element depleted leach solution comprises the original leach solution, such as ammonium sulphate, and the excess of oxalic acid. The separation is typically performed by filtration. The rare earth element recovery step is performed in the presence of excess oxalic acid. Typically the oxalic acid is used approximately 5 - 30 times in excess with respect to the stoichiometric amount.

The method comprises an oxalate recovery step, wherein the rare earth element depleted leach solution obtained from the rare earth element recovery step is contacted with calcium compound, for precipitating unused excess oxalic acid as calcium oxalate and the precipitated, solid calcium oxalate is separated from the remaining leach solution. The calcium compound is any suitable calcium compound, such as calcium hydroxide, calcium carbonate or calcium chloride. As stated above, oxalic acid is used in high excess and therefore it is extremely important to recover unused oxalic acid. This is enabled with the oxalate recovery step of the present method. In the present method the remaining leach solution obtained from oxalate recovery step can

be recycled back to the leaching step. This results in economical and environmental savings.

The method comprises an oxalic acid regeneration step, wherein the separated calcium oxalate is contacted with sulphuric acid for producing oxalic acid and precipitated calcium sulphate. Typically this step is done by digestion with sulphuric acid. Sulphuric acid is typically used 3 to 6 times in excess with respect to the stoichiometric amount required. The solution comprising sulphuric acid, calcium sulphate and oxalic acid is subjected to a solid-liquid separation and thereby a calcium sulphate cake is formed, which can be directed to a neutralization step and the solution comprising oxalic acid and sulphuric acid is subjected to oxalic acid crystallization. The oxalic acid crystallization is typically performed by evaporation-crystallization or vacuum crystallization. Typically, the solid oxalic acid obtained from oxalic acid regeneration step after the crystallization is solubilized with water and after that is recycled back to the rare earth element recovery step. This too results in economical and environmental savings.

Typically the calcium sulphate obtained from oxalic acid regeneration step is further subjected to a neutralisation step. After the final neutralization step undissolved clay, gypsum solids and water can be safely disposed together with leach solid residues and directed to tailings.

The present invention relates also to an arrangement of recovering rare earth element(s) from ion adsorption clay comprising

- a leaching unit, which is adapted for leaching the ion adsorption clay in a leach solution for producing a leach slurry,
- a solid-liquid separation unit, which is adapted for washing leach residue contained in the leach slurry with water and for separating a pregnant leach solution and solid matter from each other which are contained in the leach slurry obtained from the leaching unit,
- a concentration unit, which is adapted for separating water from the pregnant leach solution obtained from the solid-liquid separation unit, thereby producing a concentrated pregnant leach solution containing dissolved solids,
- a rare earth element recovery unit, which is adapted for contacting the concentrated pregnant leach solution obtained from the concentration unit with oxalic acid for precipitating rare earth element(s) as rare earth oxalates(s) and the precipitated rare earth oxalate(s) are separated from the rare earth element depleted leach solution,

- oxalate recovery unit, which is adapted for contacting the rare earth element depleted leach solution obtained from the rare earth element recovery step with calcium compound, such as calcium hydroxide, calcium carbonate or calcium chloride, for precipitating unused oxalic acid as calcium oxalate and the calcium oxalate is separated from the remaining leach solution,

- oxalic acid regeneration unit, which is adapted for contacting calcium oxalate obtained from the oxalic acid recovery unit with sulphuric acid for producing oxalic acid and precipitated calcium sulphate.

The same definitions as presented in connection with the description of the method apply also to the description of the arrangement.

Typically the ion adsorption clay comprises Al-silicate minerals e.g. kaolinite, K-silicate minerals e.g. microcline, Al-minerals e.g. gibbsite or any combination thereof having REE adsorbed on it. The ion adsorption clay may comprise any of the above mentioned either alone or in any combination thereof.

Typically the leaching unit is a reactor leaching unit.

Typically, in the leaching step the leach solution is sulphate solution, mixture of sulphate solutions, chloride solution, mixture of chloride solutions or a mixture of sulphate and chloride solution(s). Examples of sulphate solutions are ammonium sulphate solution, sodium sulphate solution or a mixture thereof, etc. Examples of chloride solutions are potassium chloride, sodium chloride etc.

In the arrangement, the solid-liquid separation unit typically comprises a circuit of counter-current decantation thickeners. Typically the circuit of counter-current decantation thickeners contains 2 to 10 devices, more typically 5 to 6.

The concentration unit comprises typically evaporation equipment or membrane filtration equipment, preferably reverse osmosis equipment.

Furthermore, the arrangement is typically adapted for recycling the water separated in the concentration unit for washing of solid matter in the solid liquid-separation unit.

The rare earth element recovery unit comprises excess of oxalic acid.

Furthermore, the arrangement is typically adapted for recycling leach solution obtained from oxalate recovery unit back to the leaching unit.

The arrangement is typically adapted for recycling oxalic acid, which is crystallized and solubilized with water in oxalate regeneration unit, back to the rare earth element recovery unit.

In more detail, the arrangement is typically adapted for subjecting the solution comprising sulphuric acid, calcium sulphate and oxalic acid to a solid-liquid separation and thereby forming a calcium sulphate cake, which can be directed to a neutralization step and for subjecting the solution comprising oxalic acid and sulphuric acid to oxalic acid crystallization. The oxalic acid regeneration unit comprises for oxalic acid crystallization evaporation-crystallization equipment or vacuum crystallization equipment. Typically, the arrangement is adapted for recycling the water solubilized solid oxalic acid obtained from oxalic acid regeneration unit after the crystallization, back to the rare earth element recovery unit. This too results in economical and environmental savings.

Calcium sulphate obtained from oxalic acid regeneration unit is further subjected to a neutralisation unit.

List of reference numbers

	1	leaching unit
20	2	solid-liquid separation step
	3	concentration step
	4	rare earth element precipitation
	5	S/L separation of REE precipitate
	6	oxalate precipitation step
25	7	S/L separation of oxalate solids
	8	calcium sulphate precipitation
	9	solid-liquid separation (gypsum removal)
	10	oxalic acid crystallization
	11	neutralization
30	101	ion adsorption clay
	102	ammonium sulphate
	103	leach slurry
	104	water (make-up water)
	105	leach residue (underflow from thickener)
35	106	pregnant leach solution
	107	recycled process water
	108	concentrated pregnant leach solution

	109	slurry containing rare earth elements
	110	oxalic acid
	111	rare earth element oxalate for further processing
	112	rare earth element depleted leach solution
5	113	calcium hydroxide
	114	calcium oxalate slurry
	115	remaining leach solution (regenerated)
	116	calcium oxalate
	117	sulphuric acid
10	118	calcium sulphate + oxalic acid + sulphuric acid
	119	calcium sulphate-cake
	120	oxalic acid + sulphuric acid solution (aqueous)
	121	oxalic acid (solid)
	122	recycled sulphuric acid solution
15	123	calcium hydroxide
	124	slurry to tailings

Figure 1 is a flow diagram of an example embodiment of the present method. Ion adsorption clay 101 is directed to a leaching unit 1 together with an ammonium sulphate 102 leach solution. Remaining leach solution, which is regenerated and obtained from solid-liquid separation 7, is recycled to the leaching unit 1 as a stream 115. In the leaching unit 1 rare earth elements contained in the ion adsorption clay are transferred into the leach solution thereby producing a leach slurry 103, which is directed to a solid-liquid separation step 2. Make-up water 104 and recycled process water 107 are fed to the solid-liquid separation step 2, which typically comprises a circuit of 5 to 6 counter-current decantation thickeners. In the solid-liquid separation step 2 the solid matter, i.e. leach residue contained in the leach slurry is washed with water and removed from the solid-liquid separation step 2 as a leach residue (under-flow from thickener) 105. From the solid-liquid separation step 2 is obtained a pregnant leach solution 106, from which the leach residue has been separated. The pregnant leach solution 106 is directed to a concentration step 3, wherein water is separated from the pregnant leach solution 106 and recycled back to the solid-liquid separation step 2 as recycled process water 107. The concentration step 3 typically comprises evaporation equipment or membrane filtration equipment, such as reverse osmosis equipment. A concentrated pregnant leach solution 108 is obtained from the concentration step 3 and di-

rected to rare earth element precipitation step 4. Oxalic acid 110 and recycled oxalic acid 120 are directed to the rare earth element precipitation step 4, wherein the concentrated pregnant leach solution 108 is contacted with oxalic acid 110 and 120 for precipitating rare earth element(s) as rare earth oxalate(s). Oxalic acid is used in excess. The precipitated rare earth oxalate(s) 111 are separated from a rare earth element depleted leach solution 112 in a solid-liquid separation of rare earth element precipitate step 5. The precipitated rare earth oxalate(s) 111 are directed to further processing. Steps 4 and 5 form together a rare earth element recovery step. The rare earth element depleted leach solution 112 is directed to an oxalate precipitation step 6. Calcium hydroxide 113 is fed to the oxalate precipitation step 6 and rare earth element depleted leach solution 112 obtained from the rare earth element recovery step (steps 4 and 5) is contacted with calcium hydroxide 113 for precipitating unused excess oxalic acid as calcium oxalate and thereby forming a calcium oxalate slurry 114. The obtained calcium oxalate slurry 114 is directed to solid-liquid separation step of oxalate solids 7, wherein calcium oxalate is separated from the calcium oxalate slurry thereby forming a remaining leach solution 115, which is directed back to leaching step 1. Steps 6 and 7 form together an oxalate recovery step. The obtained solid calcium oxalate 116 is directed from solid-liquid separation step of oxalate solids 7 to a calcium sulphate precipitation step 8. Sulphuric acid 117 is directed to calcium sulphate precipitation step 8, wherein the separated calcium oxalate 116 is contacted with sulphuric acid for producing a solution comprising oxalic acid, sulphuric acid and precipitated calcium sulphate 118. The mixture of oxalic acid, sulphuric acid and calcium sulphate 118 is directed to a solid-liquid separation step of calcium sulphate removal 9, wherein the solid calcium sulphate is separated from the liquid mixture of oxalic acid and sulphuric acid, typically by filtration. The obtained oxalic acid and sulphuric acid mixture 120 is directed to oxalic acid crystallization step 10 and the calcium sulphate-cake 119 is directed to a neutralization step 11. In the neutralization step 11 the natural acidity caused by the ion adsorption clay to the leach residue is neutralized with the help of calcium hydroxide 123 and the neutralized product comprising undissolved clay, gypsum residue, water and residue slurry, is directed to tailings 124. In the oxalic acid crystallization step 10 oxalic acid is crystallized by evaporation/crystallization and/or vacuum crystallization. The solid oxalic acid obtained from the oxalic acid crystallization step 10 is solubilized with water and recycled back to rare earth element precipitation step 4. The obtained sulphuric acid solution 122 is recycled

to calcium sulphate precipitation step 8. Steps 8, 9 and 10 form together an oxalic acid regeneration step.

It will be obvious to a person skilled in the art that, as the technology advances, the inventive concept can be implemented in various ways. The invention and its embodiments are not limited to the examples described above
5 but may vary within the scope of the claims.

CLAIMS

1. A method of recovering rare earth element(s) from ion adsorption clay comprising

5 - a leaching step, wherein the ion adsorption clay is leached in a leach solution for producing a leach slurry,

 - a solid-liquid separation step, wherein the leach slurry is subjected to a solid-liquid separation step for washing leach residue contained in the leach slurry with water and for separating a pregnant leach solution and the leach residue from each other,

10 - a concentration step, wherein water is separated from the pregnant leach solution thereby producing a concentrated pregnant leach solution containing the dissolved solids,

 - a rare earth element recovery step, wherein the concentrated pregnant leach solution is contacted with oxalic acid for precipitating rare earth element(s) as rare earth oxalate(s) and the precipitated rare earth oxalate(s) are separated from the rare earth element depleted leach solution,

15 - oxalate recovery step, wherein the rare earth element depleted leach solution obtained from the rare earth element recovery step is contacted with calcium compound for precipitating unused excess oxalic acid as calcium oxalate and the calcium oxalate is separated from the remaining leach solution,

20 - oxalic acid regeneration step, wherein the separated calcium oxalate is contacted with sulphuric acid for producing oxalic acid and precipitated calcium sulphate.

25 2. The method according to claim 1, wherein in the leaching step the leach solution is selected from a sulphate solution, such as ammonium sulphate solution or sodium sulphate solution; a chloride solution, such as potassium chloride or sodium chloride; and any mixture thereof, such as a mixture of sulphate solutions, a mixture of chloride solutions or a mixture of sulphate and chloride solution(s).

30 3. The method according to claim 1 or 2, wherein the leaching step is performed as reactor leaching.

 4. The method according to any one of the preceding claims, wherein the solid-liquid separation step is done in a circuit of counter-current decantation thickeners.

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5. The method according to any one of the preceding claims, wherein the concentration step is performed with evaporation or membrane filtration technology, such as reverse osmosis.

6. The method according to any one of the preceding claims, wherein the water separated in the concentration step is recycled for washing of leach residue in the solid liquid-separation step.

7. The method according to any one of the preceding claims, wherein the rare earth element recovery step is performed in the presence of excess oxalic acid.

8. The method according to any one of the preceding claims, wherein the remaining leach solution obtained from oxalate recovery step is recycled back to the leaching step.

9. The method according to any one of the preceding claims, wherein the calcium compound is calcium hydroxide, calcium carbonate or calcium chloride.

10. The method according to any one of the preceding claims, wherein the oxalic acid is crystallized in the oxalic acid regeneration step and the crystallized oxalic acid is solubilized with water and recycled back to the rare earth element recovery step.

11. The method according to any one of the preceding claims, wherein calcium sulphate obtained from oxalic acid regeneration step is further subjected to a neutralisation step.

12. The method according to any one of the preceding claims, wherein in the leaching step the leach solution is ammonium sulphate.

13. The method according to any one of the preceding claims, wherein the ion adsorption clay comprises Al-silicate minerals e.g. kaolinite, K-silicate minerals e.g. microcline, Al-minerals e.g. gibbsite or any combination thereof having REE adsorbed on it.

14. An arrangement of recovering rare earth element(s) from ion adsorption clay comprising

- a leaching unit, which is adapted for leaching the ion adsorption clay in a leach solution for producing a leach slurry,

- a solid-liquid separation unit, which is adapted for washing leach residue contained in the leach slurry with water and for separating a pregnant leach solution and solid matter from each other which are contained in the leach slurry obtained from the leaching unit,

- a concentration unit, which is adapted for separating water from the pregnant leach solution obtained from the solid-liquid separation unit, thereby producing a concentrated pregnant leach solution containing the dissolved solids,

5 - a rare earth element recovery unit, which is adapted for contacting the concentrated pregnant leach solution obtained from the concentration unit with oxalic acid for precipitating rare earth element(s) as rare earth oxalates(s) and the precipitated rare earth oxalate(s) are separated from the rare earth element depleted leach solution,

10 - oxalate recovery unit, which is adapted for contacting the rare earth element depleted leach solution obtained from the rare earth element recovery step with calcium compound, such as calcium hydroxide, calcium carbonate or calcium chloride, for precipitating unused oxalic acid as calcium oxalate and the calcium oxalate is separated from the remaining leach solution,

15 - oxalic acid regeneration unit, which is adapted for contacting calcium oxalate obtained from the oxalic acid recovery unit with sulphuric acid for producing oxalic acid and precipitated calcium sulphate.

20 15. The arrangement according to claim 14, wherein the leach solution in the leach unit is selected from a sulphate solution, such as ammonium sulphate solution or sodium sulphate solution; a chloride solution, such as potassium chloride or sodium chloride; and any mixture thereof, such as a mixture of sulphate solutions, a mixture of chloride solutions or a mixture of sulphate and chloride solution(s).

25 16. The arrangement according to claim 14 or 15, wherein the leach unit is a reactor leach unit.

 17. The arrangement according to any one of claims 14 to 16, wherein the solid-liquid separation unit comprises a circuit of counter-current decantation thickeners.

30 18. The arrangement according to claim 17, wherein the circuit of counter-current decantation thickeners comprises 2 to 10 devices, typically 5 to 6 devices.

 19. The arrangement according to any one of the preceding claims 14 to 18, wherein the concentration unit comprises evaporation equipment or
35 membrane filtration equipment, such as reverse osmosis equipment.

20. The arrangement according to any one of the preceding claims 14 to 19, wherein the arrangement is adapted for recycling the water separated in the concentration unit for washing of leach residue obtained from the solid liquid-separation unit.

5 21. The arrangement according to any one of the preceding claims 14 to 20, wherein the rare earth element recovery unit comprises excess of oxalic acid.

 22. The arrangement according to any one of the preceding claims 14 to 21, wherein the arrangement is adapted for recycling leach solution ob-
10 tained from oxalate recovery unit back to the leaching unit.

 23. The method according to any one of the preceding claims 14 to 22, wherein the arrangement is adapted for recycling oxalic acid crystallized in oxalic acid regeneration unit and solubilized with water back to the rare earth element recovery unit.

15 24. The arrangement according to any one of the preceding claims 14 to 23, wherein the arrangement is adapted for directing calcium sulphate obtained from oxalic acid regeneration unit further to a neutralisation unit.

 25. The arrangement according to any one of the preceding claims 14 to 24, wherein the leach solution in the leach unit is ammonium sulphate.

20 26. The arrangement according to any one of the preceding claims 14 to 25, wherein the ion adsorption clay comprises Al-silicate minerals e.g. kaolinite, K-silicate minerals e.g. microcline, Al-minerals e.g. gibbsite or any combination thereof having REE adsorbed on it.

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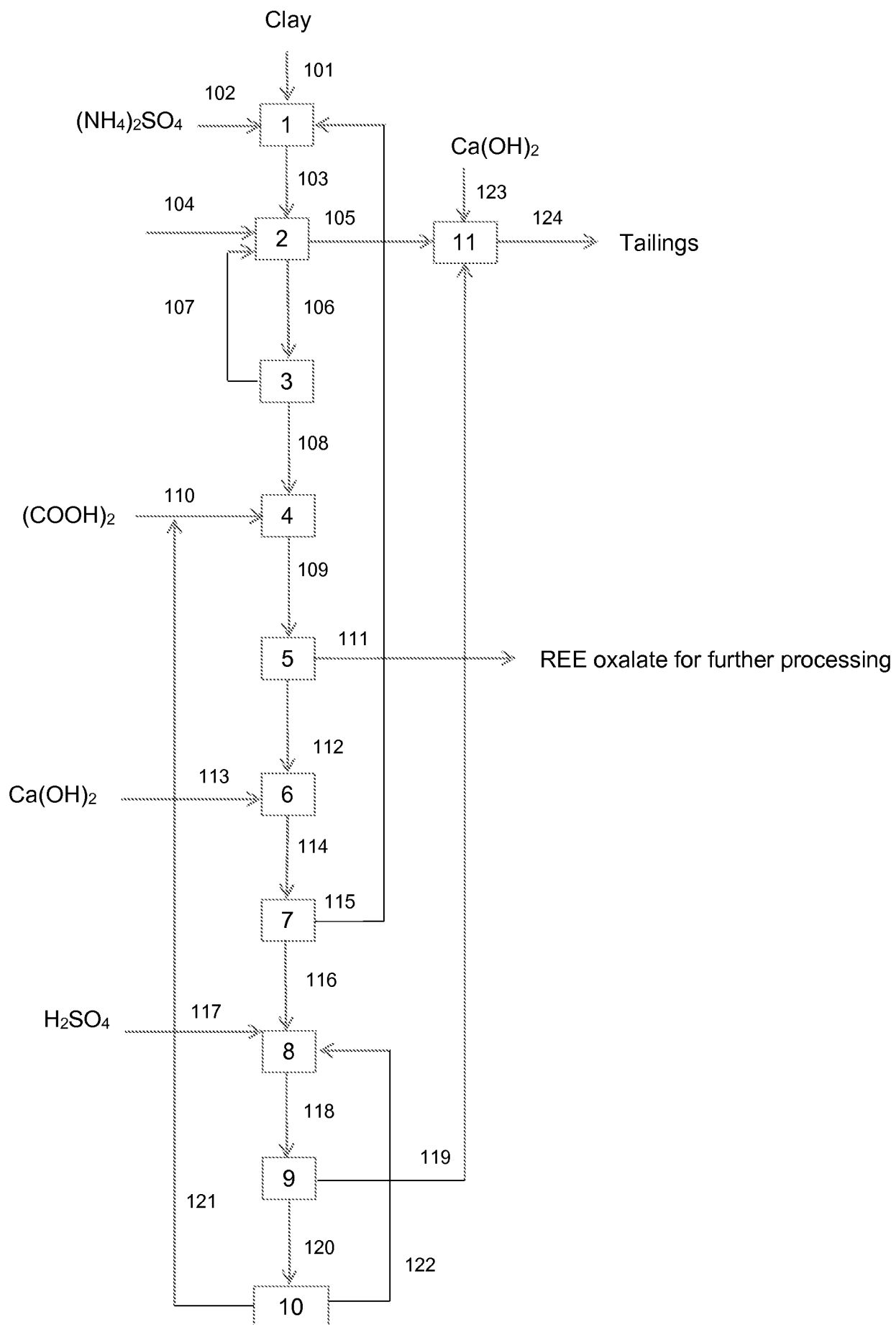


Fig. 1

INTERNATIONAL SEARCH REPORT

International application No
PCT/FI2016/050085

A. CLASSIFICATION OF SUBJECT MATTER
INV. C22B59/00
ADD.

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)
C03C C22B

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

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

EP0-Internal

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	GEORGIANA A. MOLDOVEANU ET AL: "Recovery of rare earth elements adsorbed on clay minerals: I. Desorption mechanism", HYDROMETALLURGY., vol. 117-118, 1 April 2012 (2012-04-01), pages 71-78, XP055268679, NL ISSN: 0304-386X, DOI: 10.1016/j.hydromet.2012.02.007 abstract; p. 195-7: "IV. Overview of Leaching Technologies for the Ion-Adsorption Clays" with p. 198: "Batch Leaching Tests"; p. 192: "4. Weathered Crust Elution-deposited Rare Earth Ores" -----	1-13
Y	US 2 198 527 A (CURTIN LEO P) 23 April 1940 (1940-04-23) figure; p. 1, l. 42-52; p. 2, l. 33ff ----- -/-	1-13



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents:

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Date of the actual completion of the international search

3 May 2016

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Name and mailing address of the ISA/

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INTERNATIONAL SEARCH REPORT

International application No
PCT/FI2016/050085

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	SINGH D K ET AL: "Recovery of oxalate from scrubbing solution containing rare earths and iron produced during uranium recovery from phosphoric acid", DESALINATION, ELSEVIER, AMSTERDAM, NL, vol. 232, no. 1-3, 30 November 2008 (2008-11-30), pages 37-48, XP025404945, ISSN: 0011-9164, DOI: 10.1016/J.DESAL.2007.09.015 [retrieved on 2008-09-09] abstract; "2. Experimental"	1-13
Y	Vladimiros G. Papangelakis et.al.: "RECOVERY OF RARE EARTH ELEMENTS FROM CLAY MINERALS", 4 September 2014 (2014-09-04), XP055268750, Retrieved from the Internet: URL:http://www.eurare.eu/docs/eres2014/fifthSession/VladimirosPapangelakis.pdf [retrieved on 2016-04-26] the whole document	1-13
Y	US 2014/283652 A1 (SUGITA KAORU [JP] ET AL) 25 September 2014 (2014-09-25) the whole document	1-13

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/FI2016/050085

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			JP 5545418 B2	09-07-2014
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