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— *of inventorship (Rule 4.17(iv))*

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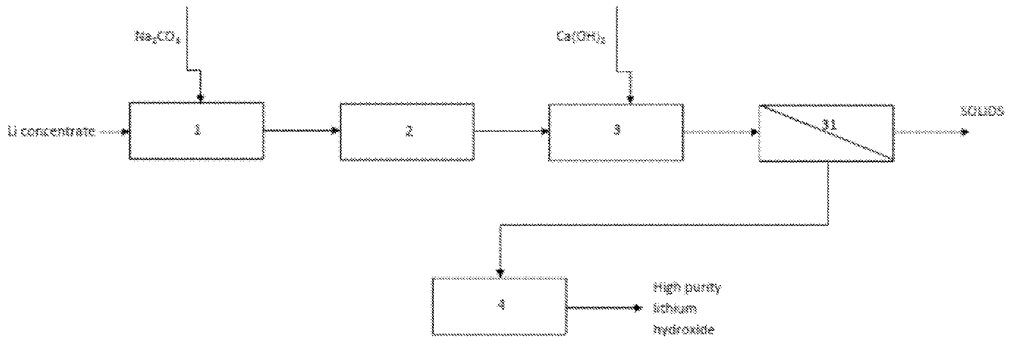
(54) Title: METHOD FOR RECOVERING LITHIUM HYDROXIDE

Fig. 1



(57) Abstract: A method and arrangement for recovering lithium hydroxide from a raw material containing lithium, which method comprises pulping (1) the raw material containing lithium in the presence of water and an alkali metal carbonate for producing a first slurry containing lithium from the raw material containing lithium. After pulping the lithiumcontaining first slurry is leached (2) for a first time thus producing a second slurry containing lithium carbonate, followed by a second leaching (3) for producing a third slurry containing lithium hydroxide. After pulping and said two leaching steps the method comprises a separation (31) of the solids from the solution. Crystals of lithium hydroxide monohydrate are then recovered by crystallizing (4).

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METHOD FOR RECOVERING LITHIUM HYDROXIDE

FIELD OF THE INVENTION

The present invention relates to a method and an arrangement for recovering lithium hydroxide.

BACKGROUND OF THE INVENTION

CN102115101 discloses a method for producing lithium carbonate from spodumene mineral by performing a sulfuric acid treatment in order to obtain lithium sulfate, followed by a step of preparing the lithium carbonate mother liquor, from which the carbonate product can be separated, and finally the lithium hydroxide is obtained from the mother liquor by adding lime to causticize said mother liquor. Also barium hydroxide is said to be useful as a causticizing hydroxide.

CN 100455512 C discloses a process for preparing lithium hydroxide monohydrate by adding sodium hydroxide to a lithium sulfate solution in order to obtain liquid lithium hydroxide, followed by cooling, filtering and separating the lithium hydroxide from the sodium sulfate, whereafter a series of recrystallization steps are performed to provide the pure lithium hydroxide monohydrate.

In CN 1214981 C a similar process is described, wherein the step of adding sodium hydroxide into the lithium sulfate solution is carried out, followed by cooling and separating to obtain the liquid lithium hydroxide. The lithium hydroxide solution is then concentrated and crystallized, whereby a coarse lithium hydroxide monohydrate product can be separated. In this publication the pure lithium hydroxide monohydrate is obtained by reacting the coarse product with barium hydroxide, followed by concentrating and crystallizing.

However, these processes all proceed via the lithium sulfate.

BRIEF DESCRIPTION OF THE INVENTION

An object of the present invention is thus to provide a method and an arrangement for recovering lithium hydroxide with high yield and high purity, without the need for multiple purification steps. In addition, the process concept is sulphate and acid free, without the formation of undesired crystallized byproducts. The objects of the invention are achieved by a method and an arrangement which is characterized by what is stated in the independent claims.

The preferred embodiments of the invention are disclosed in the dependent claims.

The present invention relates to a method for recovering lithium hydroxide from a raw material containing lithium. The method comprises the following steps of

- pulping the raw material containing lithium in the presence of water and an alkali metal carbonate for producing a first slurry containing lithium,
- leaching the first slurry containing lithium in a first leaching step at an elevated temperature for producing a second slurry containing lithium carbonate,
- leaching the second slurry or a fraction thereof in a second leaching step in an aqueous solution containing an alkali earth metal hydroxide for producing a third slurry containing lithium hydroxide,
- separating the third slurry into solids containing impurities and a solution containing lithium hydroxide by solid-liquid separation and providing a purified solution containing lithium hydroxide, and
- recovering lithium hydroxide monohydrate crystals by crystallising from the purified solution containing lithium hydroxide.

According to an embodiment of the present invention the raw material containing lithium can be any raw material from which it is desired to recover lithium. Typically the raw material containing lithium is selected from a mineral containing lithium, preferably from spodumene, petalite or lepidolite or mixtures thereof, most suitably from spodumene.

According to an embodiment of the present invention the raw material containing lithium is selected from a mineral containing lithium which has undergone heat treatment, whereby a particularly preferred material is beta-spodumene.

According to an embodiment of the present invention the first leaching solution is separated from the solids after the first leaching step, whereby only the solids are carried to the second leaching step.

According to an embodiment of the present invention the first leaching solution is separated from the solids after the first leaching step and is recycled either to the pulping step or to the first leaching step, or a fraction to each.

According to an embodiment of the present invention the solids are separated from the solution in the separation step by any suitable solid-liquid separation method, typically by thickening and/or filtering.

According to an embodiment of the present invention a separate purifying step is carried out on the solution obtained from the second solid/liquid separation step, which preferably is performed by ion exchange, typically by using cation exchange resin.

According to an embodiment of the present invention the crystallising of the lithium hydroxide monohydrate is performed by heating and cooling, or alternatively by merely concentrating the solution by heating.

According to an embodiment of the present invention the bleed solution obtained while crystallizing the lithium hydroxide monohydrate is recovered and recycled to one or more of the previous process steps, for example the pulping step, second leaching step, a separation step, and/or a point upstream of the crystallization step.

Optionally, the bleed solution obtained from the crystallization step is pretreated prior to recycling it to previous process steps, e.g. by adjusting the pH of the solution by carbonation (CO_2).

When performing a first, optional, solid-liquid separation between the leaching steps, it is possible to recover the solution used in the first leaching step, containing any excess of the leaching chemical, i.e. the alkali metal carbonate, and recycle it. This recycling, in turn, reduces the need for a separate pH adjustment in the later stages of the process, and likewise reduces the chemical consumption in the process.

According to a preferred embodiment of the present invention, when a first solid-liquid separation is carried out between the two leaching steps, the obtained solids are partly recovered and carried to a separate process for recovering pure lithium carbonate in a process comprising the following steps

- mixing the solids containing lithium into an aqueous solution to prepare a further slurry containing lithium carbonate,
- carbonating said further slurry containing lithium carbonate by using carbon dioxide for producing a solution containing lithium bicarbonate,
- separating solids from the solution containing lithium bicarbonate by solid-liquid separation, and
- recovering lithium carbonate by crystallising it from the solution containing lithium bicarbonate.

When applying this preferred embodiment, the method can also include the steps of recycling the crystallized lithium carbonate to the second hydroxide leaching step, and recycling the bleed solution obtained from the hydroxide crystallization, as described above, to one or more previous process steps, for example the pulping step, the second leaching step, a separation step and/or a point upstream of the crystallization step.

The present invention relates also to an arrangement for recovering lithium hydroxide from a raw material containing lithium according to the above method, which arrangement comprises

- a pulping unit 1 for pulping the raw material containing lithium in the presence of water and an alkali metal carbonate,
- a first leaching unit 2 for leaching a first slurry containing lithium at an elevated temperature to form a second slurry containing lithium carbonate,
- a second leaching unit 3 for leaching the second slurry containing lithium carbonate using a second leaching reagent, in order to produce a third slurry,
- a solid-liquid-separation unit 31 for separating solids from the third slurry, and
- a crystallising unit 4 for recovering lithium hydroxide monohydrate from the liquid phase obtained from the separation unit 31.

According to an embodiment of the invention the arrangement comprises also the necessary lines for carrying the solutions to be recycled to their intended units.

BRIEF DESCRIPTION OF THE DRAWINGS

In the following the invention will be described in greater detail by means of preferred embodiments with reference Figure 1, which shows a general flow diagram and arrangement of units of an embodiment of the invention, and Figure 2, which shows a general flow diagram and arrangement of units of another embodiment of the invention.

DETAILED DESCRIPTION OF THE INVENTION

An embodiment of the method of the invention, as presented schematically in Figure 1, is a method for recovering lithium hydroxide from a raw material containing lithium, which method comprises pulping 1 the raw material containing lithium in the presence of water and an alkali metal carbonate for extracting the lithium from the raw material and producing a first slurry contain-

ing lithium. A preferred alkali metal carbonate is sodium carbonate. Typically, the alkali metal carbonate is present in excess.

After pulping the first lithium-containing slurry is leached 2 for a first time for producing a second slurry containing lithium carbonate.

After an optional solid-liquid separation step 21, the lithium-containing phase (here typically the solids, or the entire second slurry) is leached 3 for a second time using a hydroxide reagent, i.e. an earth alkali metal hydroxide, followed by a separation of solids from the solution by solid-liquid separation 31 and by the preparation of lithium hydroxide -containing solution of high purity.

If a separation step is performed between the leaching steps 2,3 the liquid fraction of the first leaching solution can be recycled either to the pulping step or to the first leaching step, or a fraction to each, and the solid phase carried to the second leaching step 3.

Finally, crystals of lithium hydroxide monohydrate are recovered by crystallising 4 from the purified lithium-containing solution. The method of the invention enables production of pure lithium hydroxide monohydrate with excellent yield and purity in a continuous and simple process.

The bleed solution obtained while crystallizing 4 the lithium hydroxide monohydrate can be recovered and recycled to one or more of the previous process steps, for example the pulping step 1, second leaching step 3, a separation step 31, and/or an upstream level of the crystallization step 4.

The raw material containing lithium can be any suitable mineral containing lithium from which it is desired to recover lithium. Typically the raw material containing lithium is selected from a mineral containing lithium, such as spodumene, petalite, lepidolite or mixtures thereof. Typically the lithium-containing raw material is spodumene, preferably in the form of beta-spodumene.

According to an embodiment of the invention the method may also comprise steps for pretreating the lithium-containing raw material. If necessary, it is possible to perform a heat treatment of the lithium-containing raw material before pulping. For example, spodumene occurs in nature as α-spodumene, however it has to be converted into β-spodumene before pulping and leaching. For example, this can be performed by heating the α-spodumene or concentrate of α-spodumene to a temperature of approximately 1050°C for a suitable period of time.

The pulping 1 can be performed in any suitable vessel or reactor by contacting a feed containing lithium mineral with an alkali metal carbonate and water for producing a first slurry containing lithium.

The first leaching 2 of the first slurry containing lithium is typically performed in a suitable autoclave or series of autoclaves at an elevated temperature, for example achieved by the use of high-pressure steam. The first leaching 2 is typically performed at the temperature of 160 to 250°C, preferably 200-220°C. A typical pressure of the first leaching step 2 is 10-30bar, preferably 15-25bar. The presence of alkali metal carbonate and process conditions result in the formation of lithium carbonate and analcime solids, which can be presented in the case of spodumene and sodium carbonate with the following formula (1).

$$2\ LiAl(SiO_3)_2 + Na_2CO_3 = 2NaAl(SiO_3)_2 + Li_2CO_3 \quad (1)$$

The formed second slurry containing lithium carbonate is then routed to a second leaching step 3, performed in any suitable vessel or reactor, optionally after a liquid phase containing excess leaching solution has been separated from the lithium-containing solids. The second leaching 3 is preferably performed by using an earth alkali metal hydroxide as a leaching agent. Preferred alternatives of said hydroxide are calcium hydroxide and barium hydroxide, calcium hydroxide ($Ca(OH)_2$)being most preferred, optionally prepared by reaction of calcium oxide (CaO) in the aqueous solution. The temperature in the second leaching 3 is typically 10-100°C, preferably 20-60°C, and most suitable 20-40°C. A typical pressure for the second leaching step 3 is 1-10bar, preferably atmospheric pressure.

The presence of earth alkali metal hydroxide and process conditions result in the formation of lithium hydroxide, which can be presented in the case of analcime, lithium carbonate and calcium hydroxide with the following formula (2).

$$2NaAl(SiO_3)_2 + Li_2CO_3 + Ca(OH)_2 = 2NaAl(SiO_3)_2 + CaCO_3 + 2LiOH \quad (2)$$

After the two leaching steps 2,3 have been performed, the obtained third lithium hydroxide -containing slurry is separated 31 into a solid phase and a solution. The separation 31 can be done with any suitable solid-liquid sepa-

ration method. For example, the third slurry can be routed to a thickener, from where the overflow can be routed directly to purification and the underflow can be filtered further in order to recover all lithium hydroxide present in the solution and separate it from solid impurities, or a simple filtering technique can be used.

After the solid-liquid-separation 31 the solution containing lithium can optionally be purified 32 by using a suitable purifying method. According to an embodiment of the invention, the lithium-containing solution is purified with ion exchange in order to remove further impurities. Typically the purifying by ion exchange is performed by using cation exchange resin. The ion exchange can be performed for example by using a method disclosed in Finnish patent 121 785. Typically the purifying by ion exchange is performed by using cation exchange resin, wherein the cation exchange group is for example iminodiacetic acid (IDA) or aminophosphonic acid (APA).

After the separation 31 and optional purifying 32, lithium hydroxide monohydrate is recovered by crystallising it 4, for example by heating the purified solution in the crystallization unit to evaporate the liquid, or by recrystallizing the monohydrate from a suitable solvent.

The present invention relates also to an arrangement for recovering lithium hydroxide from a raw material containing lithium according to the method of the present invention. The referral numbers referring to Figure 1 in connection with the description of the method correspond to the referral numbers used in connection with the description of the arrangement, thus the method steps of the method correspond to the units of the arrangement. The arrangement comprises

- a pulping unit 1 for pulping the raw material containing lithium in the presence of water and an alkali metal carbonate,
- a first leaching unit 2 for leaching a first slurry containing lithium at an elevated temperature to form a second slurry containing lithium carbonate,
- a second leaching unit 3 for leaching the second slurry containing lithium carbonate, or a fraction thereof, obtained from the first leaching unit 2, in the presence of a hydroxide to produce a third slurry containing lithium hydroxide,
- a solid-liquid-separation unit 31 for separating the third slurry into a solid fraction and a solution containing lithium hydroxide, and

- a crystallising unit 4 for recovering lithium hydroxide monohydrate from the solution.

According to an embodiment of the invention, the second slurry obtained from the first leaching unit 2 is carried to an optional solid-liquid separation unit 21 for separation into solids and a liquid, before the lithium-containing fraction (here the solids) is carried to the second leaching unit 3.

In case of using such an optional S/L separation unit 21, it can be equipped with a line 211 for carrying a liquid fraction from said separation unit 21 to the pulping unit 1, and a line 212 for carrying a liquid fraction from said separation unit 21 to the first leaching unit 2.

According to an embodiment of the invention, the liquid phase separated from the solids in the separation unit 31, is carried to a separate purification unit 32 before being carried further to the crystallization unit 4.

According to an embodiment of the invention, the crystallizing unit 4 is equipped with a line 401 for carrying a liquid fraction from the crystallizing unit 4 to the pulping unit, and with a line 404 for carrying a liquid fraction from a downstream point to an upstream point of the crystallizing unit 4.

The arrangement also comprises all inlets, outlets and process instrumentation needed for carrying out the method of the invention.

REFERENCE NUMBERS

The reference numbers according to an embodiment of the present invention, as used in Figure 1 and Figure 2, are shown below (some of these units and lines being optional):

1			pulping unit
2			first leaching unit
	21		optional solid-liquid separation unit
		211	recycle line from separation unit 21 to pulping unit 1
		212	recycle line from separation unit 21 to first leaching unit 2
3			second leaching unit
	31		solid-liquid separation unit

32	purification unit (not shown in the Figures, but optionally present downstream from the S/L separation unit 31, and upstream from the crystallization unit 4)
4	crystallizing unit
401	recycle line from crystallizing unit 4 to pulping unit 1 (not shown in the drawings)
404	recycle line from a downstream point to an upstream point of the crystallizing unit 4 (not shown in the drawings)

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 but may vary within the scope of the claims.

EXAMPLE

The soda leaching was carried out on a slurry containing lithium, and obtained by pulping a beta-spodumene mineral sample. The leaching was performed in an autoclave with the presence of high-pressure steam and at a temperature of 220°C with a retention time of 1.5 h, with an initial solids content of 29.5 wt-%, a sodium to lithium excess in the feed of 10%, giving a pH of about 11.5.

The solids were separated from the obtained leached solution through filtration, and the obtained solid fraction was carried to a second leaching vessel, where leaching tests were carried out as shown in the following Table 1:

Table 1

Test	Time h	Temperature °C	pH	Li mg/L	Na mg/L	Al mg/L	Ca mg/L	Mn mg/L	Mg mg/L	P mg/L	K mg/L	Fe mg/L	Si mg/L	B mg/L	Be mg/L	S mg/L	Ti mg/L	Bi mg/L
CL1	1	38	10.9	6610	630	51	11	<1	<1	<50	30	<2	161	<2	<1	<20	<1	<5
CL1	2	35	10.9	6820	687	77	11	<1	<1	<50	31	<2	169	<2	<1	<20	<1	<5
CL2	1	22	11.4	6840	528	<5	34	<1	<1	<50	22	<2	16	<2	<1	<20	<1	<5
CL2	2	22	11.4	6870	528	8	25	<1	<1	<50	23	<2	26	<2	<1	<20	<1	<5

These tests showed that the used ~10% $Ca(OH)_2$ excess to lithium content was sufficient to provide the desired end-result, i.e. a lithium hydroxide solution with a low content of impurities. Also the temperature of 20-40°C was sufficient. The lower temperature of 22°C produced a more pure solution in general, particularly in relation to the content of aluminium and silicon. However, the calcium concentration in the solution was higher at this lower temperature compared to the concentration achieved at the higher temperatures.

CLAIMS

1. A method for recovering lithium hydroxide from a mineral containing lithium, selected from spodumene, petalite or lepidolite or mixtures thereof, wherein the method comprises

- pulping the raw material containing lithium in the presence of water and an alkali metal carbonate for producing a first slurry containing lithium,

- leaching the first slurry containing lithium in a first leaching step at an elevated temperature for producing a second slurry containing lithium carbonate,

- leaching the second slurry or a fraction thereof in a second leaching step in an aqueous solution containing an alkali earth metal hydroxide for producing a third slurry containing lithium hydroxide,

- separating the third slurry into solids and a solution containing lithium hydroxide by solid-liquid separation and providing a purified solution containing lithium hydroxide, and

- recovering lithium hydroxide monohydrate by crystallising from the purified solution containing lithium hydroxide.

2. The method according to claim 1, wherein the solution is separated from the solids in a separate solid-liquid separation step after the first leaching step, and the solids carried to the second leaching step.

3. The method according to any one of the preceding claims, wherein the solution is separated from the solids after the first leaching step, and the solution is recycled either to the pulping step or to the first leaching step, or a fraction to each.

4. The method according to any one of the preceding claims, wherein the second leaching step is carried out in an aqueous solution in the presence of either calcium hydroxide or barium hydroxide, preferably in the presence of calcium hydroxide.

5. The method according to any one of the preceding claims, wherein the second leaching steps is carried out at a temperature of 10-100°C, preferably 20-60°C, and most suitably 20-40°C.

6. The method according to any one of the preceding claims, wherein the solid-liquid separation is performed by thickening and/or filtering.

7. The method according to any one of the preceding claims, wherein a separate purification step is carried out after the separation of the third

slurry into solids and a solution, which purification step preferably is carried out by ion exchange, more by using cation exchange resin.

8. The method according to any one of the preceding claims, wherein the crystallising of the lithium hydroxide monohydrate is performed by heating the purified solution containing lithium to a temperature of approximately the boiling point of the solution.

9. The method according to any one of the preceding claims, wherein the solution obtained during the crystallization is separated from the process, and is recycled to one or more of the previous process steps, for example the pulping step, the second leaching step, a separation step, and/or a point upstream of the crystallization step.

10. An arrangement for recovering lithium carbonate from a raw material containing lithium according to the method of any one of claims 1 to 9, which arrangement comprises

- a pulping unit (1) for pulping the raw material containing lithium in the presence of water and an alkali metal carbonate,
- a first leaching unit (2) for leaching a first slurry containing lithium at an elevated temperature,
- a second leaching unit (3) for leaching a second slurry containing lithium carbonate, or a fraction thereof, in the presence of water and an alkali earth metal hydroxide,
- a solid-liquid-separation unit (31) for separating a third slurry containing lithium hydroxide into solids and a solution containing lithium, and
- a crystallising unit (4) for recovering lithium hydroxide monohydrate from a solution containing lithium.

11. The arrangement according to claim 10, wherein a further solid-liquid separation unit (21) is arranged between the first leaching unit (2) and the second leaching unit (3).

12. The arrangement according to claim 10 or 11, wherein a further solid-liquid separation unit (21) is arranged between the first leaching unit (2) and the second leaching unit (3), and a line (211) is arranged to carry a solution from the further separation unit (21) to the pulping unit (1), and/or a line (212) is arranged to carry a solution from the further separation unit (21) to the first leaching unit (2).

13. The arrangement according to any one of claims 10 to 12, wherein either solid-liquid-separation unit (21,31) comprises a thickening unit and/or a filtering unit.

14. The arrangement according to any one of the preceding claims 10 to 14, which comprises a line (401) for carrying a solution from the crystallizing unit (4) to the pulping unit (1), and/or a line (404) for carrying a solution from the crystallizing unit (4) to a point further upstream of the crystallizing unit (4), and preferably one or more lines for carrying a solution from the crystallization unit (4) to the second leaching unit (3) and/or to a separation unit (21, 31).

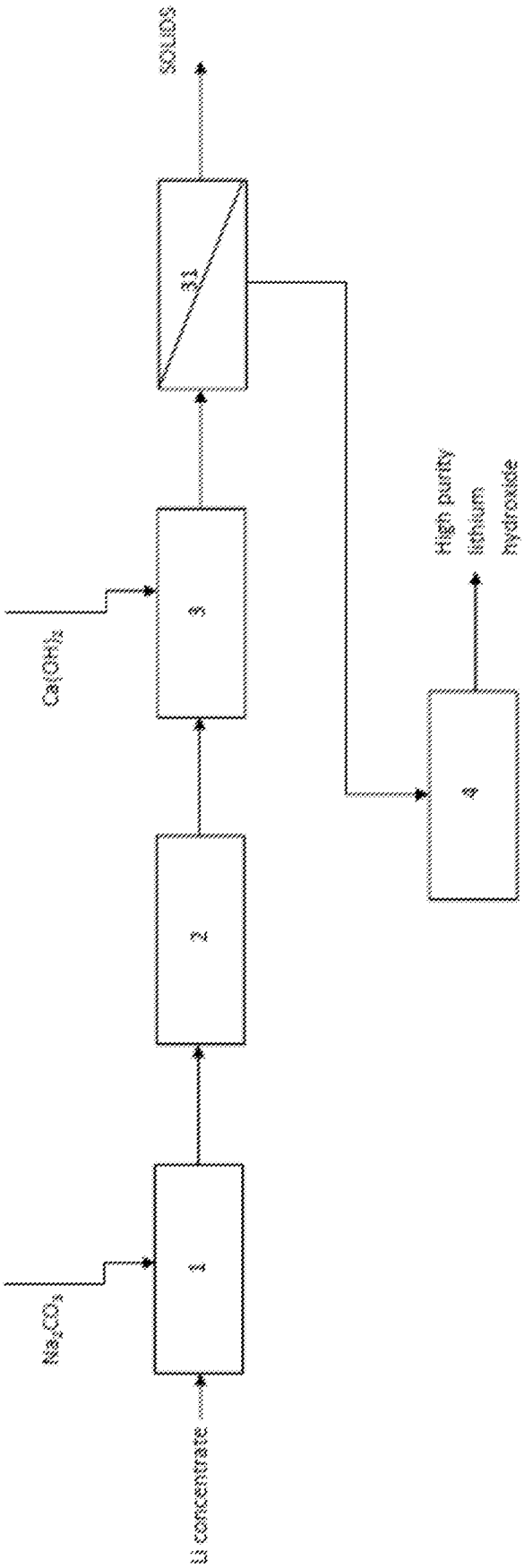
Li concentrate
Na_2CO_3
1
2
$Ca(OH)_2$
3
31
SO4/Cl
4
High purity lithium hydroxide

Fig. 1

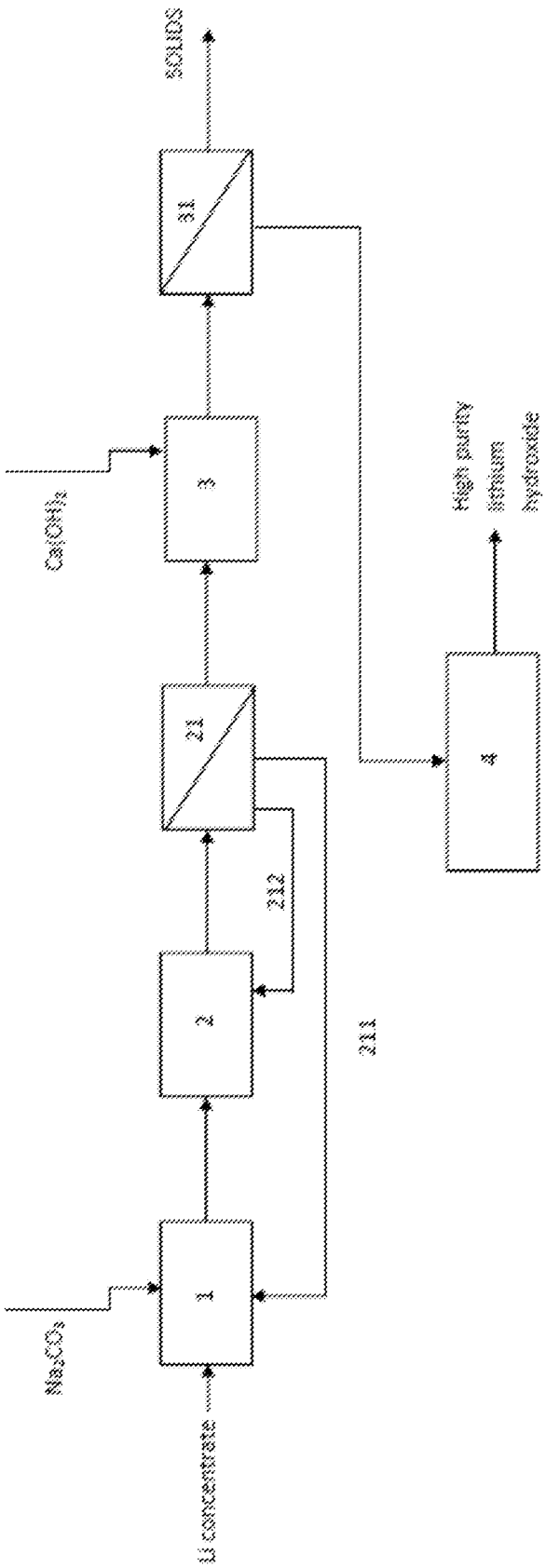
solids
31
$Ca(OH)_2$
3
High purity lithium hydroxide
4
21
212
2
211
1
Na_2CO_3
Li concentrate

Fig. 2

INTERNATIONAL SEARCH REPORT

International application No.
PCT/FI2018/050377

A. **CLASSIFICATION OF SUBJECT MATTER**

See extra sheet

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC: C01D, C22B

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

FI, SE, NO, DK

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

EPODOC, EPO-Internal full-text databases, Full-text translation databases from Asian languages, WPIAP, PRH-Internal, XPESP, COMPDX, BIOSIS

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 3343910 A (MAURICE ARCHAMBAULT et al.) 26 September 1967 (26.09.1967) column 2, lines 18-21; column 3, lines 5-67; column 6, lines 4-18; claims 1, 2	1-14
A	CN 1109104 A (XIANGXIANG ALUMINUM PLANT [CN]) 27 September 1995 (27.09.1995) & abstract [online] EPOQUENET EPODOC	1-14
A	US 3073673 A (CHUBB PHILIP A) 15 January 1963 (15.01.1963) claim 1; figure 1	1-14

☒ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

* Special categories of cited documents:
"A" document defining the general state of the art which is not considered to be of particular relevance
"E" earlier application or patent but published on or after the international filing date
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"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed
"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
27 August 2018 (27.08.2018)	30 August 2018 (30.08.2018)

Name and mailing address of the ISA/FI
Finnish Patent and Registration Office
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INTERNATIONAL SEARCH REPORT

International application No.
PCT/FI2018/050377

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	CHOUBEY P. K. et al. Advance review on the exploitation of the prominent energy-storage element: Lithium. Part I: From mineral and brine resources. In: MINERALS ENGINEERING OXFORD, GB: Elsevier Ltd., 2016-01-28, Vol. 89, pages 119-137, ISSN 0892-6875, section 2.5 Existing commercial process for lithium recovery from minerals; section 3.2 Pre-concentration/pre-treatment; figures 10, 15	1-14

INTERNATIONAL SEARCH REPORT

International application No.
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CLASSIFICATION OF SUBJECT MATTER

IPC
C01D 15/02 (2006.01)
C22B 3/04 (2006.01)
C22B 3/20 (2006.01)
C01D 7/00 (2006.01)

INTERNATIONAL SEARCH REPORT
Information on Patent Family Members

International application No.
PCT/FI2018/050377

Patent document cited in search report	Publication date	Patent family members(s)	Publication date
US 3343910 A	26/09/1967	BE 627542 A GB 1052747 A	
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US 3073673 A	15/01/1963	None	