



(51) International Patent Classification:

C22B 26/12 (2006.01) C22B 3/26 (2006.01)  
C22B 26/00 (2006.01)

(21) International Application Number:

PCT/US2020/062855

(22) International Filing Date:

02 December 2020 (02.12.2020)

(25) Filing Language:

English

(26) Publication Language:

English

(71) Applicant: **U.S. BORAX INC.** [US/US]; 251 Little Falls Drive, Wilmington, Delaware 19808 (US).

(72) Inventors: **PATWARDHAN, Amit**; c/- U.S. Borax Inc., 251 Little Falls Drive, Wilmington, Delaware 19808 (US). **SHINDE, Sandip**; c/- U.S. Borax Inc., 251 Little Falls Drive, Wilmington, Delaware 19808 (US). **WATTENBARGER, Allen**; c/- U.S. Borax Inc., 251 Little Falls Drive, Wilmington, Delaware 19808 (US). **SUASNABAR,**

**Daniel**; c/- U.S. Borax Inc., 251 Little Falls Drive, Wilmington, DE 19808 (US).

(74) Agent: **WOLFFE, Susan A.**; BANNER WITCOFF, 1100 13th St. NW, Suite 1200, Washington, District of Columbia 20005 (US).

(81) Designated States (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DJ, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IR, IS, IT, JO, JP, KE, KG, KH, KN, KP, KR, KW, KZ, LA, LC, LK, LR, LS, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SA, SC, SD, SE, SG, SK, SL, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, WS, ZA, ZM, ZW.

(84) Designated States (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, GH, GM, KE, LR, LS, MW, MZ, NA, RW, SD, SL, ST, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ,

(54) Title: A LITHIUM EXTRACTION PROCESS AND APPARATUS

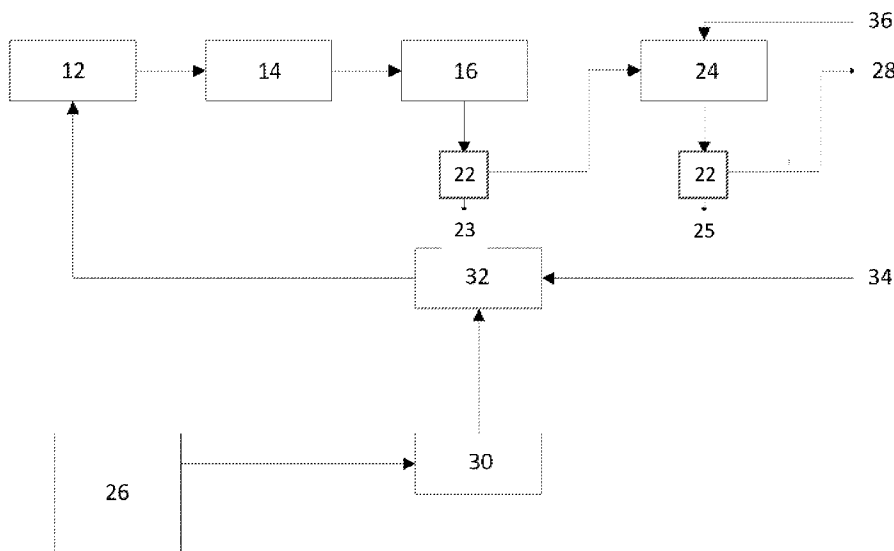


Figure 1

(57) Abstract: A method of extracting lithium from a lithium-bearing material including: (i) mixing the lithium-bearing material, gypsum, a sulfur-containing material, and a calcium-containing material and forming a feed mixture having a predetermined composition; (ii) roasting the mixture and forming a roasted mixture including a water-soluble lithium compound; and (iii) leaching lithium from the water-soluble lithium compound and forming a lithium-containing leachate by mixing the aqueous solution and the water-soluble lithium compound.



TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, KM, ML, MR, NE, SN, TD, TG).

**Published:**

— *with international search report (Art. 21(3))*

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## A LITHIUM EXTRACTION PROCESS AND APPARATUS

FIELD OF INVENTION

The invention relates to a process and an apparatus for  
5 extracting lithium from a lithium-bearing material.

In particular, although by no means exclusively, the  
invention relates to a process and an apparatus for  
extracting lithium from low grade lithium-bearing material  
10 such as waste material from borates mining or clay  
formations.

BACKGROUND

Lithium is used to make batteries for a variety of  
15 applications including electric cars, cameras and mobile  
phones.

Lithium is obtained by either extracting lithium-  
containing salts from underground brine reservoirs or  
20 mining lithium-containing rock.

One example of lithium-containing rock is in deposits in  
borates mines, with lithium being in waste rock and  
tailings generated from the borates mining and recovery  
25 process.

Low value gangue material typically ends up in a  
tailings dam or in a stacked heap. The tailings and  
stacked heaps have low concentrations of lithium that  
30 cannot be extracted economically at the present time.  
The lithium in the tailings and stacked heaps, whilst  
low grade, is a potential asset that may be unlocked  
economically later with current technology or with

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improving technology. The amounts of tailings and waste rock generated during mining can be significant and, hence, the potential lithium value can be significant.

5 Lithium is also present, typically in low concentrations, in clay formations and, to date, it has been challenging to extract lithium from these formations in an economically viable way.

10 There are a number of known processes for extracting lithium from lithium-containing materials.

However, it has been challenging to extract lithium in a practical and economic way from low grade lithium bearing material such as tailings, waste rock and clay formations described above.

It would be desirable for a process to extract lithium from low grade lithium-bearing material.

20 The above description is not an admission of the common general knowledge in Australia or elsewhere.

#### SUMMARY OF INVENTION

25 The present invention provides a process for extracting lithium from lithium-bearing material, particularly low grade lithium-bearing waste material.

The lithium-bearing material may be a sediment-hosted deposit. The sediment-hosted deposit may be waste tailings obtained from an industrial processing plant such as a boric acid processing plant or a borates mine. The

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sediment-hosted deposit may comprise lithium bearing clay minerals.

5 The lithium bearing clay minerals may be processed or treated clay for example clay minerals found in the waste material from a processing plant which may have been processed, for example, by roasting.

10 The lithium bearing clay minerals may be virgin clay such as untreated or natural clay, for example obtained from clay formations.

Examples of clay minerals include smectites such as hectorite and/or montmorillonite, Bigadic clays, and  
15 lithium bearing illite with or without lithium zeolites.

The lithium-bearing material may be material in which lithium is associated with high concentrations of sodium, aluminum, silicon and/or boron. Typically, the lithium-  
20 bearing material comprises 8-32 wt% of sodium, aluminium, silicon, potassium and/or boron per kg of lithium-bearing material. Suitably, the lithium-bearing material is a boron-containing ore.

25 The expression "low grade" refers to a lithium concentration ranging from 1-3 g/kg of lithium-bearing material.

The present invention provides a method of extracting  
30 lithium from a lithium-bearing material including:

- (i) mixing the lithium-bearing material, gypsum, a sulfur-containing material, and a calcium-

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containing material and forming a feed mixture having a predetermined composition;

(ii) roasting the mixture and forming a roasted mixture including a water-soluble lithium compound; and

(iii) leaching lithium from the water-soluble lithium compound and forming a lithium-containing leachate by mixing the aqueous solution and the water-soluble lithium compound.

The present invention also provides a method of extracting lithium from a lithium-bearing material including:

(i) mixing the lithium-bearing material, gypsum, a sulfur-containing material, and a calcium-containing material and forming a feed mixture having a predetermined composition;

(ii) supplying the mixture to a roaster;

(iii) roasting the mixture in the roaster and forming a roasted mixture including a water-soluble lithium compound;

(iv) supplying the water-soluble lithium compound to a leach tank;

(v) supplying an aqueous solution to the leach tank; and

(vi) leaching lithium from the water-soluble lithium compound and forming a lithium-containing leachate by mixing the aqueous solution and the water-soluble lithium compound in the leach tank.

One advantage of the present invention is that it provides a lithium extraction process that can extract value from waste material, for example waste rock and

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tailings, generated from a variety of industrial processes including, but not limited to, borates mining.

Another advantage of the present invention is that it provides a lithium extraction process that reduces the operating cost of the process. The roasting step in a known lithium extraction method is recognised as a key driving factor for operational costs. The present invention replaces part of the gypsum used in the known roasting step with a functionally-equivalent substance (i.e. the sulfur-containing material including elemental sulfur or an alkali metal sulfate, such as sodium or potassium sulfate) that can be generated on-site or in-situ to reduce the operating cost.

A further advantage of the present invention is that it provides a process that uses an environmentally benign substance, water, in the extraction process instead of highly acidic solutions. This is achieved by a roasting step in which Li-silicate from clays is converted into  $\text{Li}_2\text{SO}_4$  which is water soluble.

The sulfur-containing material may be either or a combination of an alkali metal sulfate and elemental sulfur.

The alkali metal sulfate may be either or a combination of sodium sulfate and potassium sulfate. Suitably, the alkali metal sulfate is sodium sulfate.

The alkali metal sulfate may be obtained from an effluent waste stream of a processing plant.

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Suitably, the alkali metal sulfate is obtained from boric acid plant liquor or tailings pond.

5 The calcium-containing material may be either or a combination of calcium carbonate such as limestone or dolomite, and lime. The calcium-containing material may be substituted with magnesium carbonate.

10 The mixing step may involve mixing wet lithium-bearing material with gypsum, a sulfur-containing material, and a calcium-containing material and forming a mixture. Suitably, the wet lithium-bearing material has a water content ranging from 30-60 wt%. More suitably, the wet lithium-bearing material has a water content ranging from  
15 40-50wt%.

The mixing step may involve adding an aqueous solution to the lithium-bearing material having a water content of less than 30 wt% to form the wet lithium-bearing material.

20 The mixing step may form a mixture having a composition in which the gypsum: sulfur-containing material ratio is at least 1:1 (e.g. 1kg gypsum: 1kg sodium sulfate).

25 Suitably, the amount of gypsum: sulfur-containing material ratio is at least 2:1 (e.g. 2kg gypsum: 1kg sodium sulfate).

30 Suitably, the gypsum: sulfur-containing material ratio is at least 3:1 (e.g. 3kg gypsum: 1kg sodium sulfate).

Even more suitably, the gypsum: sulfur-containing material ratio is 7:3 (e.g. 7kg gypsum: 3kg sodium sulfate).



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The applicant has discovered that < 30% substitution of gypsum with a sulfur-containing material delivers lithium recoveries comparable to gypsum-only mixtures but at  
5 reduced operating costs.

The applicant discovered that complete substitution of gypsum with sodium sulfate resulted in a lithium recovery of less than 25%.

10

The mixing step may form a mixture having a predetermined composition comprising lithium-bearing material: calcium-containing material: gypsum: sulfur-containing material at a ratio of lithium-bearing material (1): calcium-  
15 containing material (0.4-0.8): gypsum (0.3-0.5): sulfur-containing material (0.1-0.3). Suitably, the mixing step forms a mixture comprising lithium-bearing material: calcium-containing material: gypsum: sulfur-containing material at a ratio of 100:45-75:20-50:10-25.

20

The above ratios for calcium-containing material may apply to calcium carbonate such as limestone or dolomite, and lime.

25 The above sulfur-containing material ratios are particularly suitable for sodium sulfate. In this respect, a skilled person would understand that adjustments may have to be made to the ratios if a different sulfur-containing material, for example potassium sulfate, is  
30 used.

The method may include a granulating step to process the mixture into granules. Suitably, the granules have a mean

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diameter less than 15 mm. More suitably, the granules have a mean diameter less than 10 mm. Even more suitably, the granules have a mean diameter less than 5 mm.

5 The roasting step may be performed at a roasting temperature ranging from 800-1,000°C.

Suitably, the roasting step is performed at a roasting temperature ranging from 850-950°C.

10

More suitably, the roasting step is performed at a roasting temperature ranging from 857-925°C. Even more suitably, the roasting step is performed at a roasting temperature of 900°C.

15

The roasting step may be performed for a roasting time period ranging from 0.5 hrs to 2 hours. Suitably, the roasting step is performed for a roasting time period of 1 hour.

20

The roasting time period is the time period during which the mixture is exposed to the roasting temperature. This roasting time period may be different to the residence time of the mixture in a kiln or furnace where the mixture  
25 may not be exposed to the roasting temperature for its entire residence time in the kiln or furnace. For example, the mixture may be exposed to a varying temperature profile as it is conveyed through a kiln. In this example, the roasting time period when the mixture is conveyed to a  
30 location in the kiln where it is exposed to the roasting temperature.

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The roasting step may be performed at a roasting temperature ranging from 800-1,000°C for a roasting time period ranging from 0.5 hrs to 2 hours.

- 5 When the mixture is roasted in a kiln, the roasting step is performed at a roasting temperature of 850°C for a roasting time period of 1 hour.

10 When the mixture is roasted in a crucible placed in a furnace, the roasting step is performed at a roasting temperature of 900°C for a roasting time period of 1 hour.

15 Sulfur-containing material including sodium, potassium and calcium sulfates may be generated during the roasting step. It can also be appreciated that unreacted sulfur-containing material may also be present in the roasted material. As this material is an ingredient of the roasting recipe, recycling this material back into the mixing tank reduces feed material costs.

20

As such, the method may include a step of adding sulfur-containing material from the roasting step to the mixing step.

- 25 The method may include a step of separating sulfur-containing material from the roasted material. Suitably, the step of separating sulfur-containing material from the roasted material involves crystallisation.

- 30 The method may include a step of adding sulfur-containing material from the crystallisation step to the mixing step.

The water-soluble lithium compound may be lithium sulfate.

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The method may include a step of crushing the water-soluble lithium compound before the leaching step.

Suitably, the crushing step involves reducing the particle size of the water-soluble lithium compound to 1,000–3,000  $\mu\text{m}$  (1–3 mm).

The leaching step may include adding an aqueous solution to the roasted mixture to form a slurry having a solids content ranging from 20–50wt%, suitably, a solids content ranging from 25–45wt%, more suitably, a solids content ranging from 30–40wt%.

The method may include counter-current leaching of the lithium from the water-soluble lithium compound. Suitably, the method may include two or more counter-current leaching steps.

In this specification, the aqueous solution used in the leaching step may have a pH ranging from 6.5–7.5. Suitably, the pH of the aqueous solution is 7.

It can be appreciated that while it is preferred that aqueous solution used in the process has a pH of 7, the process can also utilise water from a variety of sources which may contain minerals or substances that causes the pH to deviate from 7 by  $\pm 0.5$ .

The leaching step may be performed at a temperature less than 60°C. Suitably, the leaching step is performed at a temperature less than 50°C. More suitably, the leaching step is performed at a temperature ranging from 20–40°C.

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The method may include filtering the slurry to remove undissolved solids such as calcium carbonate and clay. Suitably, the filtering step generates a lithium-containing leachate having a lithium concentration of at least 2,000ppm.

The method may include concentrating the leachate.

The concentrating step may involve evaporating part of the leachate to form a concentrated leachate having a lithium concentration of at least 3,000ppm.

Suitably, the concentrating step involves evaporating part of the leachate to form a concentrated leachate having a lithium concentration of at least 4,000ppm.

More suitably, the concentrating step involves evaporating part of the leachate to form a concentrated leachate having a lithium concentration of at least 4,500ppm.

The concentrating step may result in the formation of impurities including calcium and sodium salts and/or particulate matter including thenardite, glaserite, glauberite, or anhydrite.

The method may include filtering the concentrated lithium-containing leachate to remove the impurities.

The filtered concentrated lithium-containing leachate may be processed via a series of steps to form lithium carbonate. The applicant has developed a process of forming lithium carbonate from the filtered concentrated lithium-containing leachate which is the subject of a US

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patent application filed on the same day as the present application by the same applicant, the disclosure of which is incorporated in its entirety.

5 The method may include recycling alkali metal sulfate (e.g. sodium sulfate) formed during the roasting step to supplement the sulfur-containing material in the feed material.

10 The invention also provides an apparatus to perform the previously described method.

In one form, the invention provides an apparatus for extracting lithium from a lithium-bearing material

15 comprising:

- (i) a mixing tank configured to receive and mix lithium-bearing material with gypsum, a sulfur-containing material, and a calcium-containing material and form a feed mixture having a  
20 predetermined composition;
- (ii) a roaster configured to receive and roast the mixture and form a roasted mixture including a water-soluble lithium compound; and
- (iii) a leach tank configured to form a lithium-  
25 containing leachate from the water-soluble lithium compound using an aqueous solution.

The apparatus may be located near or connected to a source of lithium-bearing material and be configured to receive  
30 this material.

The mixing tank may be connected to a tailings pond to receive the lithium-bearing material.

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The mixing tank may be connected to a boric acid plant to receive at least part of the sulfur-containing material such as an alkali metal sulfate. Suitably, the mixing tank is configured to receive an alkali metal sulfate from boric acid plant liquor or tailings pond. More suitably, the mixing tank is connected to a crystalliser for separating sodium sulfate from waste material generated by the boric acid plant.

5  
10

The apparatus may include a granulator to process the mixture from the mixing tank into granules. Suitably, the granules have a mean diameter less than 15 mm. More suitably, the granules have a mean diameter less than 10 mm. Even more suitably, the granules have a mean diameter less than 5 mm.

15

The mixing tank may be configured to receive alkali metal sulfate generated during the roasting step.

20

The apparatus may include a crystalliser to separate the sulfur-containing material from the roasted mixture from the roaster. This allows the sulfur-containing material to be recycled back to the mixing tank.

25

The crystalliser may be configured to perform flash crystallisation. Flash crystallisation is a process which enables the solution to reach its crystallisation temperature rapidly.

30

The mixing tank may be connected to the crystalliser to receive the separated sulfur-containing material.

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The roaster may be a kiln.

The apparatus may include a crusher to reduce the particle size of the roasted mixture from the roaster to 1,000-  
5 3,000  $\mu\text{m}$  (1-3 mm).

The leach tank may form part of a counter-current leaching circuit.

10 The apparatus may comprise three leach tanks arranged in series.

The leach tank may include a filter to generate a lithium-containing leachate having a lithium concentration of at  
15 least 2,000ppm.

The apparatus may include an evaporator to evaporate at least part of the leachate from the leach tank to form a concentrated leachate having a lithium concentration of at  
20 least 3,000ppm.

The evaporator may include a filter to remove impurities from the concentrated lithium-containing leachate.

#### 25 BRIEF DESCRIPTION OF DRAWINGS

The invention is hereinafter described by way of example only with reference to the accompanying Figure 1, which is a flow diagram of the process according to one form of the  
30 invention.



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DETAILED DESCRIPTION

The applicant has carried out research and development work on a known method of extracting lithium from lithium-bearing deposit. The known method includes  
5 roasting the deposit with calcium carbonate and gypsum and acid leaching the roasted material to extract the lithium.

Disadvantages of this process include the use externally  
10 sourced reagents including environmentally hazardous acid. In addition, acid leaching may not be adapted to extract lithium from material containing low concentrations of lithium because of the relatively unselective nature of acid leaching compared to water leaching.

15 The applicant has discovered that a gypsum/sulfur-containing material mixture can reduce operating costs without losing the efficiency associated with traditional calcium carbonate: gypsum recipes in generating water-  
20 soluble lithium compounds. The applicant also discovered that a gypsum/alkali metal sulfate mixture provides a more efficient roasting process compared to a mixture that excludes gypsum.

25 The applicant also realised that boric acid plants produce sodium sulfate as a waste product which can be routed to the apparatus of the present invention to reduce the amount of sodium sulfate that have to be purchased or synthesised for the present invention.

30 The applicant also realised that the roasting step may produce in-situ sodium sulfate which can be routed to the apparatus of the present invention to further reduce the

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amount of sodium sulfate that have to be purchased or synthesised for the present invention.

5 As a result of these realisations, the applicant has developed an apparatus for extracting lithium from a lithium-bearing material in accordance with the present invention. The apparatus 10 as shown in Figure 1 comprises a mixing tank 12, a roaster in the form of calciner 14, and a leach tank 16.

10

The apparatus 10 is located near or connected to a source of lithium-bearing material and is configured to receive this material. Examples of suitable lithium-bearing material sources include a tailings pond of a borates mine or clay formations.

15

Suitably, the apparatus 10 is also located near or connected to a source of an alkali metal sulfate such as sodium sulfate.

20

The sodium sulfate and lithium-bearing material may be obtained from the same source. For example, the apparatus 10 may be connected to the tailings pond of a boric acid processing plant to receive the lithium-bearing gangue and connected to a sodium sulfate-containing effluent stream of the same plant to receive sodium sulfate.

25

The apparatus may include a hopper to hold the lithium-bearing gangue. In this embodiment, the hopper is located over a vibrating pan (or screw) that feeds into an impact mill to comminute the gangue. The impact mill in turn feeds the comminuted gangue onto a 40 mesh vibratory screen that is positioned over a surge bin.

30

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The apparatus may include a surge bin to store the classified gangue before it is fed into the mixing tank 12.

5

The other feed material including a calcium-containing material such as calcium carbonate and gypsum can also be stored in separate bins before being fed into the mixing tank 12.

10

The mixing tank 12 is configured to receive inputs of lithium-bearing material, a sulfur-containing material such as an alkali metal sulfate or elemental sulfur, gypsum and a calcium-containing material such as calcium carbonate and mix these materials in specific proportions according to a predetermined roasting recipe, for example the recipes described in Tables 1 and 2 below.

15

A product outlet of the mixing tank 12 may be connected to a pelletiser for granulating the mixture.

20

The pelletizer may in turn be connected to a dryer to dry the granulated mixture.

25

A calciner 14 is configured to receive and roast the granulated mixture to convert the lithium-bearing material into a water-soluble lithium compound such as lithium sulfate.

30

A product outlet of the calciner 14 is connected to a feed inlet of a leach tank 16. In some embodiments, the calciner 14 may be connected to a cooler to cool the roasted material before it is directed to the leach tank

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16. In these embodiments, the cooler may be connected to a crusher to reduce the particle size of the roasted mixture to 1,000-3,000  $\mu\text{m}$  (1-3 mm).

5 The crushed compound may be stored in a surge bin to hold the roasted material before it is directed to the leach tank 16.

The leach tank 16 further includes a fluid inlet 18 to  
10 receive water.

The leach tank 16 is configured to enable countercurrent flow of the lithium-bearing feed material and water during leaching of the water-soluble lithium compound to form a  
15 lithium-containing leachate.

The applicant discovered that countercurrent flow of the lithium bearing material and water during the leaching process, along with a number of operating parameters,  
20 optimised the extraction of lithium from the lithium bearing material.

The leach tank 16 may be temperature controlled to enable the leaching process to be performed at a predetermined  
25 temperature.

The leach tank 16 may include a filter 22 to remove any undissolved solids formed during the leaching process. Other suitable solid-liquid separation techniques may be  
30 used to remove undissolved solids formed during the leaching process, including centrifugation.

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The leach tank 16 includes a product outlet which is connected to an inlet of evaporator 24.

5 A surge tank may be connected to the leach tank 16 to hold the filtered leachate before it is directed to the evaporator 24.

The evaporator 24 receives and concentrates the leachate from the surge tank or directly from the leach tank.  
10 Impurities such as thenardite, glaserite, glauberite, and anhydrite may precipitate during the evaporation process. The leach tank 16 may include another filter 22 to remove the precipitates from the leachate to form a concentrated leachate 28 which can be directed downstream for further  
15 processing or stored for later use.

In operation, the apparatus according to the invention is connected to a borates processing plant 26. Feed material comprising lithium-containing waste material, for example  
20 from a tailings pond or a stacked heap from the plant, is directed to a flotation circuit 30 to remove some of the non-lithium bearing material from the waste material. The lithium-bearing concentrate exiting the flotation circuit is then directed towards a dryer 32 to reduce the water  
25 content of the concentrate, preferably to 40-50 wt% before it is stored in a bin. Suitable examples of lithium-bearing material include waste lithium-bearing clay minerals include smectites such as hectorite and/or montmorillonite, Bigadic clays, and lithium bearing illite  
30 with or without lithium zeolites that have been subjected to a variety of treatment steps such as roasting in the processing plant. It was discovered that feeding lithium-bearing material having a water content ranging from 40-50

- 20 -

wt% enhanced the roasting step because the water content improves the granulation of the feed material prior to roasting.

5 Separate bins may be used to store a sulfur-containing material such as elemental sulfur or an alkali metal sulfate, gypsum and calcium-containing material such as calcium carbonate. The alkali metal sulfate may be sourced from an effluent stream typically containing sodium  
10 sulfate, from the same plant.

These bins are connected to the mixing tank 12 which receives these materials in specific proportions to form a mixture that will eventually be processed via a series of  
15 intermediate steps to form a concentrated lithium-containing solution of at least 4,000 ppm. The gypsum and calcium-containing material are typically sourced externally. The calcium-containing material can be substituted with magnesium carbonate, dolomite or lime.  
20

The sulfur-containing material is used to replace part of the gypsum in the roasting recipe. This reduces the need to commercially source gypsum and may repurpose the waste output from the borates processing plant. Importantly,  
25 this arrangement allows commercial value to be extracted from waste products from a boric acid processing plant which would otherwise have been discarded and reduces the reliance on externally sourced reagents. It also improves tailings pond management.  
30

The gangue material may be directed to an impact mill and passed through a classification screen to obtain -40 mesh particles before being fed to the mixing tank 12.

The mixing tank 12 feeds the various components based on a preselected recipe to form a mixture having a lithium-bearing material: calcium carbonate: gypsum: sodium sulfate ratio of 100:30-40:20:20. Another suitable recipe has a gypsum: sodium sulfate ratio of 7:3.

The mixture may be processed in a pelletizer to form granules before being directed to a calciner 14 for roasting at a temperature ranging from 857-925°C for about one hour.

In one embodiment, the mixture may be mixed with water to facilitate the granulation process. Alternatively, a wet mixture having a water content of 40-50 wt% may be fed directly into the pelletizer. If necessary, the granulated mixture may be dried in a dryer before being sent to the calciner 14.

Examples of suitable roasting recipes wherein the lithium-bearing material is waste lithium-bearing clay material are reproduced in Tables 1 and 2 below.

Table 1: Examples of predetermined roasting recipes for lithium-bearing clay including sodium sulfate

Recipe No	Clay	Limestone	Gypsum	Sodium Sulfate
1	100	45	40	10
2	100	45	45	15

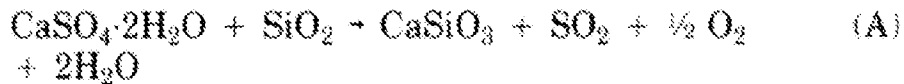
In Table 1, the mixture is roasted at a roasting temperature of 900°C for a roasting time period of 60 minutes.

Table 2: Examples of predetermined roasting recipes for lithium-bearing clay including sodium sulfate and/or elemental sulfur.

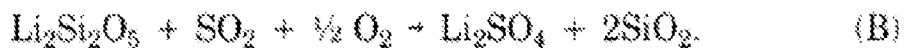
Recipe No	Clay	Limestone/Lime	Gypsum	Sodium Sulfate	Elemental S	% Li Recovery	STD. DEV	System recovery %
1	100	45	50	0		85.2	2.1	76.7
2	100	60	30	0	10	84.4	1.3	75.9
3	100	70	0	0	15	70.0	5.4	63.0
4	100	65	20	10	10	78.7	1.2	70.8
5	100	75	0	25	10	73.5	2.8	66.1
6	100	45	40	10		78.0	2.3	70.2

5 This roasting step converts the lithium bearing material into a water-soluble form for a subsequent water leaching step.

10 Representative chemical equations of the roasting process are set out below (Crocker.L Lithium and its recovery from low-grade nevada clays [Report]. - [s.l.]: Bureau of Mines, 1988).



and



15 Reaction (B) above produces sodium sulfate and/or potassium sulfate which can be recovered and returned to the mixing tank 12 to supplement the source of alkali metal sulfate.

20 The roasted material is fed into the leach tank 16 in countercurrent flow to a leaching solution of water to leach lithium from the formed water-soluble lithium



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compounds. The solids content of the roasted material in the leach tank ranges from 20-40 wt%, suitably about 30 wt%. The roasted material may be directed into a cooler before being fed to the leach tank.

5

The water used in the leaching step is ideally at a pH of 7. However, it can vary between 6.5-7.5 depending on the water source.

10

In some embodiments, the method may include a step of crushing the roasted material, including the water-soluble lithium compound, before the leaching step to enhance the leaching process. Suitably, the crushed material has a particle size ranging from 1,000-3,000  $\mu\text{m}$ .

15

The leaching step is performed at a temperature of less than 50°C. The applicant determined that a leaching temperature of about 50°C optimised the leaching efficiency in view of the inverse relationship of solubility with temperature of lithium sulfate.

20

During the leaching step, any undissolved solids such as calcium carbonate and clay are removed by filter 23. At this stage, the leachate typically has a lithium concentration of at least 2,000ppm.

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The filtered leachate is then directed to an evaporator 24 to be concentrated.

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During the evaporating step, impurities in the form of calcium and sodium salts and particulate matter including any one of more of thenardite, glaserite, glauberite, and anhydrite may be formed. These impurities 25 are removed

- 24 -

by filter 22 to form a lithium-containing leachate 28 having a concentration of at least 4,500ppm. This leachate may be further processed downstream via a series of steps to form lithium carbonate or stored for other uses.

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One of these steps involves crystallisation of the lithium-containing leachate to remove further impurities from the solution. In one embodiment, the waste material obtained from the crystallisation step is returned to the flotation circuit 30 via stream 34 to recover lithium from the crystallisation step impurities.

Another step during the production of lithium carbonate is a lithium carbonate precipitation step which generates a filtrate which can be recycled back to the evaporator via stream 36.

In the claims which follow and in the preceding description of the invention, except where the context requires otherwise due to express language or necessary implication, the word "comprise" or variations such as "comprises" or "comprising" is used in an inclusive sense, i.e. to specify the presence of the stated features but not to preclude the presence or addition of further features in various embodiments of the invention.

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CLAIMS:

1. A method of extracting lithium from a lithium-bearing material including:
  - 5 (i) mixing the lithium-bearing material, gypsum, a sulfur-containing material, and a calcium-containing material and forming a feed mixture having a predetermined composition;
  - (ii) roasting the mixture and forming a roasted mixture
  - 10 (iii) leaching lithium from the water-soluble lithium compound and forming a lithium-containing leachate by mixing the aqueous solution and the water-soluble lithium compound.
- 15 2. The method according to claim 1, wherein the sulfur-containing material is either or a combination of an alkali metal sulfate and elemental sulfur.
- 20 3. The method according to claim 2, wherein the alkali metal sulfate is either or a combination of sodium sulfate and potassium sulfate.
4. The method according to claim 1, wherein the calcium-
- 25 containing material is either or a combination of calcium carbonate and lime.
5. The method according to claim 1, wherein the mixing
- step involves mixing lithium-bearing material having a
- 30 water content ranging from 30-60 wt% with the gypsum, the sulfur-containing material, and the calcium-containing material.

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6. The method according to claim 1, wherein the mixing step involves adding an aqueous solution to a lithium-bearing material having a water content of less than 30 wt% to form the wet lithium-bearing material.
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7. The method according to claim 1, wherein the mixing step forms a mixture in which the gypsum: sulfur-containing material ratio is at least 1:1.
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8. The method according to claim 1, wherein the mixing step forms a mixture having a predetermined composition comprising lithium-bearing material: calcium-containing material: gypsum: sulfur-containing material at a ratio of lithium-bearing material (1): calcium-containing material (0.4-0.8): gypsum (0.3-0.5): sulfur-containing material (0.1-0.3).
- 15
9. The method according to claim 1, including a granulating step to process the mixture into granules having a mean diameter less than 15 mm.
- 20
10. The method according to claim 1, wherein the roasting step is performed at a roasting temperature ranging from 800-1,000°C for a roasting time period ranging from 0.5 hrs to 2 hours.
- 25
11. The method according to claim 1, including a step of separating sulfur-containing material from the roasted material using crystallisation.
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12. The method according to claim 11, including a step of adding the sulfur-containing material obtained from the crystallisation step to the mixing step.

- 27 -

13. The method according to claim 1, including a step of reducing the particle size of the water-soluble lithium compound to 1,000-3,000  $\mu\text{m}$  (1-3 mm).

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14. The method according to claim 1, wherein the leaching step includes adding an aqueous solution to the roasted mixture to form a slurry having a solids content ranging from 20-50wt%.

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15. The method according to claim 14, wherein the aqueous solution used in the leaching step has a pH ranging from 6.5-7.5.

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16. The method according to claim 14, including filtering the slurry to generate a lithium-containing leachate having a lithium concentration of at least 2,000ppm.

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17. The method according to claim 1, including evaporating part of the leachate to form a concentrated leachate having a lithium concentration of at least 3,000ppm.

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18. The method according to claim 17, including filtering the concentrated lithium-containing leachate to remove impurities.

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19. The method according to claim 1, including recycling alkali metal sulfate formed during the roasting step to supplement the sulfur-containing material in the feedmixture.

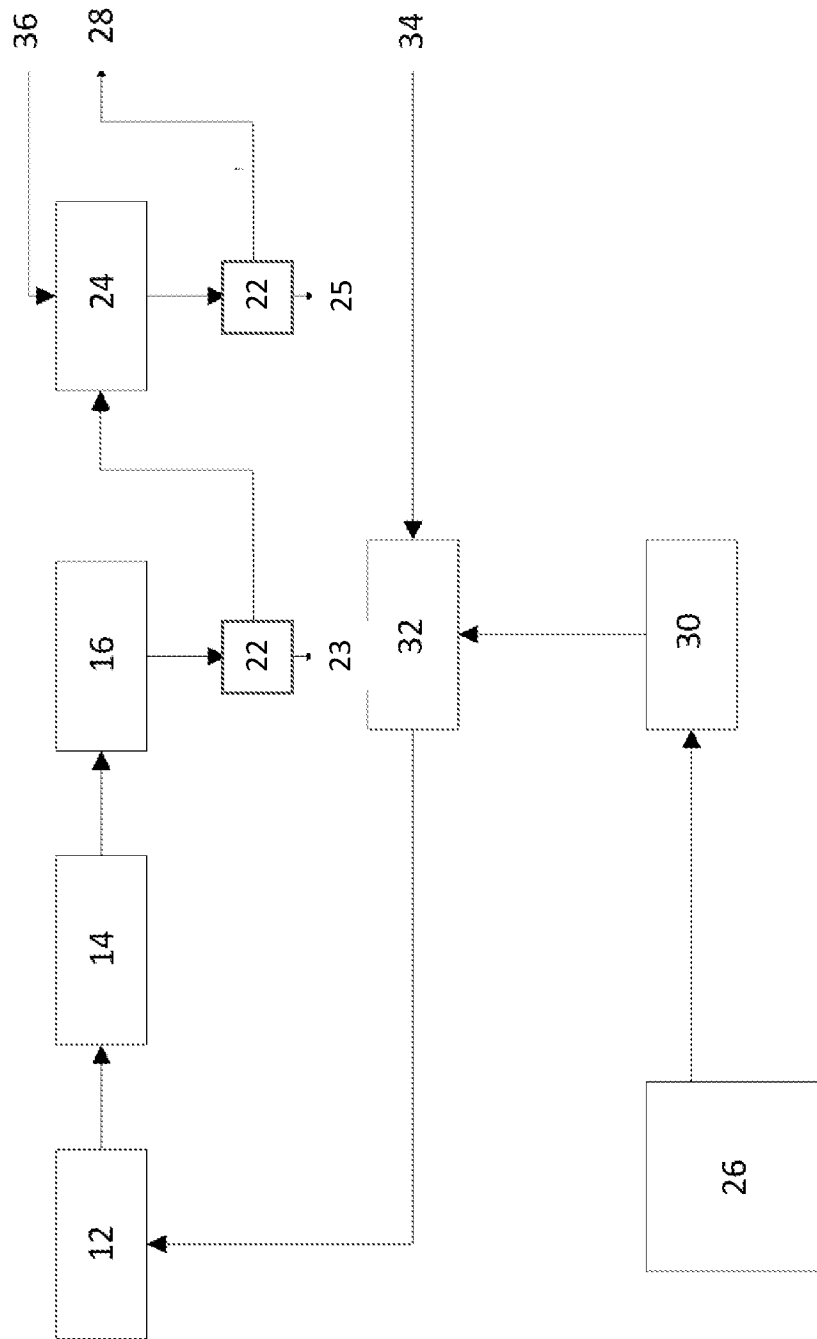


Figure 1

## INTERNATIONAL SEARCH REPORT

International application No.

PCT/US 20/62855

A. CLASSIFICATION OF SUBJECT MATTER  
 IPC - C22B 26/12; C22B 26/00; C22B 3/26 (2021.01)  
 CPC - C22B 26/12; C22B 3/0005; C22B 3/44; C22B 7/006

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)  
 See Search History document

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

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

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	~ Pittuck et al. "Technical Report on the Feasibility Study for the Sonora Lithium Project, Mexico." Ausenco Services Pty Ltd, 25 Jan. 2018.	1-19
A	US 8,431,005 B1 (Zbranek et al.) 30 April 2013 (30.04.2013) Entire document	1-19
A	US 2013/0206607 A1 (Kojima et al.) 15 August 2013 (15.08.2013) Entire document	1-19
A	US 2006/0171869 A1 (Anovitz et al.) 3 August 2006 (03.08.2006) Entire document	1-19
A	US 3,131,022 A (Archambault) 28 April 1964 (28.04.1964) Entire document	1-19

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

"D" document cited by the applicant in the international application

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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

22 January 2021

Date of mailing of the international search report

02 MAR 2021

Name and mailing address of the ISA/US

Mail Stop PCT, Attn: ISA/US, Commissioner for Patents  
 P.O. Box 1450, Alexandria, Virginia 22313-1450

Facsimile No. 571-273-8300

Authorized officer

Lee Young

Telephone No. PCT Helpdesk: 571-272-4300