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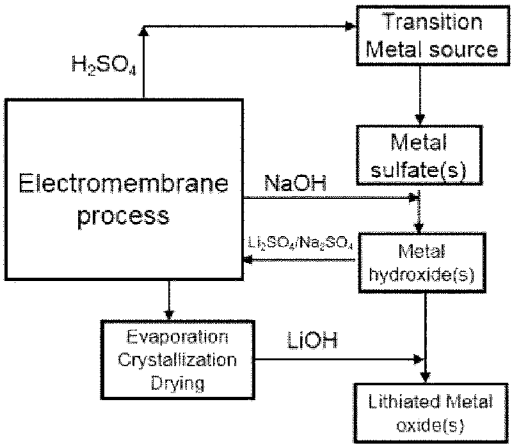
(54) Title: PROCESSES FOR PREPARING HYDROXIDES AND OXIDES OF VARIOUS METALS AND DERIVATIVES THEREOF



(57) Abrégé/Abstract:

There are provided processes for preparing a metal hydroxide comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum, the process comprising: reacting a metal sulfate comprising with lithium hydroxide, sodium hydroxide and/or potassium hydroxide and optionally a chelating agent in order to obtain a solid comprising the metal hydroxide and a liquid comprising lithium sulfate, sodium sulfate and/or potassium sulfate; separating the liquid and the solid from one another to obtain the metal hydroxide; submitting the liquid to an electromembrane process for converting the lithium sulfate, sodium sulfate and/or potassium sulfate into lithium hydroxide, sodium hydroxide and/or potassium hydroxide respectively; reusing the sodium hydroxide and/or lithium hydroxide obtained by the electromembrane process for reacting with the metal sulfate and/or with the metal hydroxide.

ABSTRACT

There are provided processes for preparing a metal hydroxide comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum, the process comprising: reacting a metal sulfate comprising with lithium hydroxide, sodium hydroxide and/or potassium hydroxide and optionally a chelating agent in order to obtain a solid comprising the metal hydroxide and a liquid comprising lithium sulfate, sodium sulfate and/or potassium sulfate; separating the liquid and the solid from one another to obtain the metal hydroxide; submitting the liquid to an electromembrane process for converting the lithium sulfate, sodium sulfate and/or potassium sulfate into lithium hydroxide, sodium hydroxide and/or potassium hydroxide respectively; reusing the sodium hydroxide and/or lithium hydroxide obtained by the electromembrane process for reacting with the metal sulfate and/or with the metal hydroxide.

PROCESSES FOR PREPARING HYDROXIDES AND OXIDES OF VARIOUS METALS AND DERIVATIVES THEREOF

CROSS-REFERENCE TO RELATES APPLICATIONS

[0001] The present disclosure claims priority to US application No. 62/590,260 filed and November 22, 2017; and to US application No. 62/735,013 filed and September 21, 2018.

TECHNICAL FIELD

[0002] The present disclosure relates to improvements in the field of processes for preparing metal hydroxides and metal oxides that contain at least one metal chosen from nickel, cobalt, manganese, lithium and aluminum. For example, such material can be useful in the manufacture of cathode materials for ion batteries.

BACKGROUND OF THE DISCLOSURE

[0003] Processes for preparing nickel-cobalt-manganese hydroxides, nickel-cobalt-aluminum hydroxides, lithium-cobalt hydroxides, nickel-cobalt-manganese oxyhydroxides, nickel-cobalt-aluminum oxyhydroxides, lithium-cobalt oxyhydroxides, nickel-cobalt-manganese oxides, nickel-cobalt-aluminum oxides and lithium-cobalt oxides are known. However, processes known for example lead to high costs in the production of such hydroxides and oxides as well as consumption of various chemicals.

[0004] There is thus a need for at least an alternative process for preparing such hydroxides or oxides.

SUMMARY OF THE DISCLOSURE

[0005] Therefore according to an aspect of the present disclosure, there is provided a process for preparing a metal hydroxide comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum, the process comprising:

reacting a metal sulfate comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum with lithium hydroxide and optionally a chelating agent in order to obtain a solid comprising the metal hydroxide and a liquid comprising lithium sulfate;

separating the liquid and the solid from one another to obtain the metal hydroxide;

submitting the liquid comprising lithium sulfate to an electromembrane process for converting the lithium sulfate into lithium hydroxide; and

reusing the lithium hydroxide obtained by the electromembrane process for reacting with the metal sulfate.

[0006] According to another aspect, there is provided a process for preparing a metal oxide comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum, the process comprising:

reacting a metal sulfate comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum with lithium hydroxide and optionally a chelating agent to obtain a solid comprising a metal hydroxide comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum, and a liquid comprising lithium sulfate;

separating the liquid and the solid from one another to obtain the metal hydroxide;

submitting the liquid comprising lithium sulfate to an electromembrane process for converting the lithium sulfate into lithium hydroxide; and

reusing at least a first portion of the lithium hydroxide obtained by the electromembrane process for reacting with the metal sulfate;

reacting at least a second portion of the lithium hydroxide obtained by the electromembrane process with the obtained metal hydroxide to obtain a mixture of metal hydroxides; and

roasting the mixture of metal hydroxides to obtain the metal oxide.

[0007] According to another aspect of the present disclosure, there is provided a process for preparing a metal hydroxide comprising at least one metal chosen from nickel, cobalt, manganese, lithium and aluminum, the process comprising:

reacting a metal sulfate comprising at least one metal chosen from nickel, cobalt, manganese, lithium and aluminum with a base and optionally a chelating agent in order to obtain a solid comprising the metal hydroxide and a liquid comprising lithium sulfate;

separating the liquid and the solid from one another to obtain the metal hydroxide;

submitting the liquid comprising lithium sulfate to an electromembrane process for converting the lithium sulfate into lithium hydroxide; and

reusing the lithium hydroxide obtained by the electromembrane process for reacting with the metal sulfate.

[0008] According to another aspect, there is provided a process for preparing a metal oxide comprising at least one metal chosen from nickel, cobalt, manganese, lithium and aluminum, the process comprising:

reacting a metal sulfate comprising at least one metal chosen from nickel, cobalt, manganese, lithium and aluminum with a base and optionally a chelating agent to obtain a solid comprising a metal hydroxide at least one metal chosen from nickel, cobalt, manganese, lithium and aluminum, and a liquid comprising lithium sulfate;

separating the liquid and the solid from one another to obtain the metal hydroxide;

submitting the liquid comprising lithium sulfate to an electromembrane process for converting the lithium sulfate into lithium hydroxide; and

reusing at least a first portion of the lithium hydroxide obtained by the electromembrane process for reacting with the metal sulfate;

reacting at least a second portion of the lithium hydroxide obtained by the electromembrane process with the obtained metal hydroxide to obtain a mixture of metal hydroxides; and

roasting the mixture of metal hydroxides to obtain the metal oxide.

[0009] According to an aspect of the present disclosure, there is provided a process for preparing a metal hydroxide comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum, the process comprising:

reacting a metal sulfate and/or a metal nitrate comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum with lithium hydroxide, sodium hydroxide and/or potassium hydroxide and optionally a chelating agent in order to obtain a solid comprising the metal hydroxide and a liquid comprising at least one of lithium sulfate, lithium nitrate, sodium sulfate, sodium nitrate, potassium sulfate, potassium nitrate;

separating the liquid and the solid from one another to obtain the metal hydroxide;

submitting the liquid comprising at least one of lithium sulfate, lithium nitrate, sodium sulfate, sodium nitrate, potassium sulfate and potassium nitrate to an electromembrane process for converting the least one of lithium sulfate, lithium nitrate, sodium sulfate, sodium nitrate, potassium sulfate, potassium nitrate, into at least one of least one of lithium hydroxide, sodium hydroxide, potassium hydroxide; and

reusing the at least one of lithium hydroxide, sodium hydroxide and potassium hydroxide obtained by the electromembrane process for reacting with the metal sulfate and/or metal nitrate.

[0010] According to another aspect, there is provided a process for preparing a metal oxide comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum, the process comprising:

reacting a metal sulfate and/or a metal nitrate comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum with lithium hydroxide, sodium hydroxide and/or potassium hydroxide and optionally a chelating agent to obtain a solid comprising a metal hydroxide comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum, and a liquid comprising at least one of lithium sulfate, lithium nitrate, sodium sulfate, sodium nitrate, potassium sulfate and potassium nitrate;

separating the liquid and the solid from one another to obtain the metal hydroxide;

submitting the liquid comprising at least one of lithium sulfate, lithium nitrate, sodium sulfate, sodium nitrate, potassium sulfate and potassium nitrate to an electromembrane process for converting the at least one of lithium sulfate, lithium nitrate, sodium sulfate, sodium nitrate, potassium sulfate and potassium nitrate into at least one of least one of lithium hydroxide, sodium hydroxide and potassium hydroxide; and

reusing at least a first portion of the at least one of lithium hydroxide, sodium hydroxide and potassium hydroxide obtained by the electromembrane process for reacting with the metal sulfate and/or the metal nitrate;

reacting at least a second portion of the at least one of lithium hydroxide, sodium hydroxide and potassium hydroxide obtained by the electromembrane process with the obtained metal hydroxide to obtain a mixture of metal hydroxides; and

roasting the mixture of metal hydroxides to obtain the metal oxide.

[0011] According to another aspect of the present disclosure, there is provided a process for preparing a metal hydroxide comprising at least one metal chosen from nickel, cobalt, manganese, lithium and aluminum, the process comprising:

reacting a metal sulfate and/or a metal nitrate comprising at least one metal chosen from nickel, cobalt, manganese, lithium and aluminum with a base and optionally a chelating agent in order to obtain a solid comprising the metal hydroxide and a liquid comprising at least one of lithium sulfate, lithium nitrate, sodium sulfate, sodium nitrate, potassium sulfate and potassium nitrate;

separating the liquid and the solid from one another to obtain the metal hydroxide;

submitting the liquid comprising at least one of lithium sulfate, lithium nitrate, sodium sulfate, sodium nitrate, potassium sulfate and potassium nitrate to an electromembrane process for converting the least one of lithium sulfate, lithium nitrate, sodium sulfate, sodium nitrate, potassium sulfate and potassium nitrate into at least one of lithium hydroxide, sodium hydroxide and potassium hydroxide; and

reusing the at least one of lithium hydroxide, sodium hydroxide and potassium hydroxide obtained by the electromembrane process for reacting with the metal sulfate and/or the metal nitrate.

[0012] According to another aspect, there is provided a process for preparing a metal oxide comprising at least one metal chosen from nickel, cobalt, manganese, lithium and aluminum, the process comprising:

reacting a metal sulfate and/or a metal nitrate comprising at least one metal chosen from nickel, cobalt, manganese, lithium and aluminum with a base and optionally a chelating agent to obtain a solid comprising a metal hydroxide comprising at least one metal chosen from nickel, cobalt, manganese, lithium and aluminum, and a liquid comprising at least one of lithium sulfate, lithium nitrate, sodium sulfate, sodium nitrate, potassium sulfate and potassium nitrate;

separating the liquid and the solid from one another to obtain the metal hydroxide;

submitting the liquid comprising the at least one of lithium sulfate, lithium nitrate, sodium sulfate, sodium nitrate, potassium sulfate and potassium nitrate to an electromembrane process for converting the at least one of lithium sulfate, lithium nitrate, sodium sulfate, sodium nitrate, potassium sulfate and potassium nitrate into at least one of lithium hydroxide, sodium hydroxide and potassium hydroxide; and reusing at least a first portion of the at least one of lithium hydroxide, sodium hydroxide and potassium hydroxide obtained by the electromembrane process for reacting with the metal sulfate and/or metal nitrate;

reacting at least a second portion of the at least one of lithium hydroxide, sodium hydroxide and potassium hydroxide obtained by the electromembrane process with the obtained metal hydroxide to obtain a mixture of metal hydroxides; and

roasting the mixture of metal hydroxides to obtain the metal oxide.

[0013] According to another aspect of the present disclosure, there is provided a process for preparing a metal hydroxide comprising at least one metal chosen from nickel, cobalt, manganese, lithium and aluminum, the process comprising:

reacting a first metal sulfate and/or a first metal nitrate comprising at least one metal chosen from nickel, cobalt, manganese, lithium and aluminum with a base comprising a second metal and optionally a chelating agent in order to obtain a solid comprising the metal hydroxide and a liquid comprising at least one of a second metal sulfate and a second metal nitrate;

separating the liquid and the solid from one another to obtain the metal hydroxide;

submitting the liquid comprising at least one of the second metal sulfate and the second metal nitrate to an electromembrane process for converting at least one of the second metal sulfate and the second metal nitrate into a second metal hydroxide; and

reusing the second metal hydroxide obtained by the electromembrane process for reacting with the first metal sulfate and/or the first metal nitrate.

[0014] According to another aspect, there is provided a process for preparing a metal oxide comprising at least one metal chosen from nickel, cobalt, manganese, lithium and aluminum, the process comprising:

reacting a first metal sulfate and/or a first metal nitrate comprising at least one metal chosen from nickel, cobalt, manganese, lithium and aluminum with a base comprising a second metal and optionally a chelating agent to obtain a solid comprising a metal hydroxide comprising at least one metal chosen from nickel, cobalt, manganese, lithium and aluminum, and a liquid comprising at least one of a second metal sulfate and a second metal nitrate;

separating the liquid and the solid from one another to obtain the metal hydroxide;

submitting the liquid comprising the at least one of a second metal sulfate and a second metal nitrate to an electromembrane process for converting the at least one of a second metal sulfate and a second metal nitrate into a second metal hydroxide; and

reusing at least a first portion of the second metal hydroxide obtained by the electromembrane process for reacting with the first metal sulfate and/or the first metal nitrate;

reacting at least a second portion of the second metal hydroxide obtained by the electromembrane process with the obtained metal hydroxide to obtain a mixture of metal hydroxides; and

roasting the mixture of metal hydroxides to obtain the metal oxide.

[0015] According to another aspect, there is provided a process for preparing a metal hydroxide comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum, the process comprising:

reacting a metal sulfate and/or a metal nitrate comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum with lithium hydroxide, sodium hydroxide and/or potassium hydroxide and optionally a chelating agent in order to obtain a solid comprising the metal hydroxide and a liquid comprising at least one of lithium sulfate, lithium nitrate, sodium sulfate, sodium nitrate, potassium sulfate and potassium nitrate;

separating the liquid and the solid from one another to obtain the metal hydroxide;

submitting the liquid comprising at least one of lithium sulfate, lithium nitrate, sodium sulfate, sodium nitrate to an electromembrane process for converting the least one of lithium sulfate, lithium nitrate, sodium sulfate, sodium nitrate, potassium sulfate and potassium nitrate into at least one of least one of lithium hydroxide, sodium hydroxide and potassium hydroxide; and

reusing the at least one of lithium hydroxide, sodium hydroxide and potassium hydroxide obtained by the electromembrane process for reacting with the metal sulfate and/or metal nitrate.

[0016] According to another aspect there is provided a process for preparing a metal hydroxide comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum, the process comprising:

reacting a metal sulfate comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum with lithium hydroxide, sodium hydroxide and/or potassium hydroxide and optionally a chelating agent in order to obtain a solid comprising the metal hydroxide and a liquid comprising at least one of lithium sulfate, sodium sulfate and potassium sulfate;

separating the liquid and the solid from one another to obtain the metal hydroxide;

submitting the liquid comprising at least one of lithium sulfate, sodium sulfate and potassium sulfate to an electromembrane process for converting the least one of lithium sulfate, sodium sulfate and potassium sulfate into at least one of least one of lithium hydroxide, sodium hydroxide and potassium hydroxide; and

reusing the at least one of lithium hydroxide, sodium hydroxide and potassium hydroxide obtained by the electromembrane process for reacting with the metal sulfate.

[0017] According to another aspect there is provided a process a process for preparing a metal hydroxide comprising at least one metal chosen from nickel, cobalt, manganese, lithium and aluminum, the process comprising:

reacting a metal sulfate and/or a metal nitrate comprising at least one metal chosen from nickel, cobalt, manganese, lithium and aluminum with a base chosen from LiOH, NaOH, KOH, RbOH, CsOH, $Mg(OH)_2$, $Ca(OH)_2$, $Sr(OH)_2$, or $Ba(OH)_2$ and optionally a chelating agent in order to obtain a solid comprising the metal hydroxide and a liquid comprising at least one of Li_2SO_4 Na_2SO_4, K_2SO_4, Rb_2SO_4, Cs_2SO_4, $MgSO_4$, $CaSO_4$, $SrSO_4$, $BaSO_4$, $LiNO_3$ $NaNO_3$, KNO_3, $RbNO_3$, $CsNO_3$, $Mg(NO_3)_2$, $Ca(NO_3)_2$, $Sr(NO_3)_2$ and $Ba(NO_3)_2$,

separating the liquid and the solid from one another to obtain the metal hydroxide;

submitting the liquid comprising at least one of Li_2SO_4 Na_2SO_4, K_2SO_4, Rb_2SO_4, Cs_2SO_4, $MgSO_4$, $CaSO_4$, $SrSO_4$, $BaSO_4$, $LiNO_3$ $NaNO_3$, KNO_3, $RbNO_3$, $CsNO_3$, $Mg(NO_3)_2$, $Ca(NO_3)_2$, $Sr(NO_3)_2$ and $Ba(NO_3)_2$ to an electromembrane process for converting the least one of Li_2SO_4 Na_2SO_4, K_2SO_4, Rb_2SO_4, Cs_2SO_4, $MgSO_4$, $CaSO_4$, $SrSO_4$, $BaSO_4$, $LiNO_3$ $NaNO_3$, KNO_3, $RbNO_3$, $CsNO_3$, $Mg(NO_3)_2$, $Ca(NO_3)_2$, $Sr(NO_3)_2$ and $Ba(NO_3)_2$ into at least one of LiOH, NaOH, KOH, RbOH, CsOH, $Mg(OH)_2$, $Ca(OH)_2$, $Sr(OH)_2$, and $Ba(OH)_2$; and

reusing the at least one of LiOH, NaOH, KOH, RbOH, CsOH, $Mg(OH)_2$, $Ca(OH)_2$, $Sr(OH)_2$, and $Ba(OH)_2$ obtained by the electromembrane process for reacting with the metal sulfate and/or the metal nitrate.

[0018] According to another aspect, there is provided a process for preparing a metal oxide comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum, said process comprising:

reacting a metal sulfate and/or a metal nitrate comprising at least one metal chosen from nickel, cobalt, manganese, lithium and aluminum with a base chosen from LiOH, NaOH, KOH, RbOH, CsOH, $Mg(OH)_2$, $Ca(OH)_2$, $Sr(OH)_2$, or $Ba(OH)_2$ and optionally a chelating agent in order to obtain a solid comprising the metal hydroxide and a liquid comprising at least one of Li_2SO_4 Na_2SO_4, K_2SO_4, Rb_2SO_4, Cs_2SO_4, $MgSO_4$, $CaSO_4$, $SrSO_4$, $BaSO_4$, $LiNO_3$ $NaNO_3$, KNO_3, $RbNO_3$, $CsNO_3$, $Mg(NO_3)_2$, $Ca(NO_3)_2$, $Sr(NO_3)_2$ and $Ba(NO_3)_2$,

separating said liquid and said solid from one another to obtain said metal hydroxide;

submitting said liquid comprising at least one of Li_2SO_4 Na_2SO_4, K_2SO_4, Rb_2SO_4, Cs_2SO_4, $MgSO_4$, $CaSO_4$, $SrSO_4$, $BaSO_4$, $LiNO_3$ $NaNO_3$, KNO_3, $RbNO_3$, $CsNO_3$, $Mg(NO_3)_2$, $Ca(NO_3)_2$, $Sr(NO_3)_2$ and $Ba(NO_3)_2$ to an electromembrane process for converting at least one of Li_2SO_4 Na_2SO_4, K_2SO_4, Rb_2SO_4, Cs_2SO_4, $MgSO_4$, $CaSO_4$, $SrSO_4$, $BaSO_4$, $LiNO_3$ $NaNO_3$, KNO_3, $RbNO_3$, $CsNO_3$, $Mg(NO_3)_2$, $Ca(NO_3)_2$, $Sr(NO_3)_2$ and $Ba(NO_3)_2$ into at least one of LiOH, NaOH, KOH, RbOH, CsOH, $Mg(OH)_2$, $Ca(OH)_2$, $Sr(OH)_2$, or $Ba(OH)_2$; and

reusing at least a first portion of said at least one of LiOH, NaOH, KOH, RbOH, CsOH, $Mg(OH)_2$, $Ca(OH)_2$, $Sr(OH)_2$, or $Ba(OH)_2$ obtained by said electromembrane process for reacting with said metal sulfate;

reacting at least a second portion of said at least one of LiOH, NaOH, KOH, RbOH, CsOH, $Mg(OH)_2$, $Ca(OH)_2$, $Sr(OH)_2$, or $Ba(OH)_2$ obtained by said electromembrane process with said obtained metal hydroxide to obtain a mixture of metal hydroxides; and

roasting said mixture of metal hydroxides to obtain said metal oxide.

[0019] According to an aspect of the present disclosure, there is provided a process for preparing a metal carbonate comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum, the process comprising:

reacting a metal sulfate and/or a metal nitrate comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum with lithium carbonate, sodium carbonate and/or potassium carbonate and optionally a chelating agent in order to obtain a solid comprising the metal carbonate and a liquid comprising at least one of lithium sulfate, lithium nitrate, sodium sulfate, sodium nitrate, potassium sulfate, potassium nitrate;

separating the liquid and the solid from one another to obtain the metal carbonate;

submitting the liquid comprising at least one of lithium sulfate, lithium nitrate, sodium sulfate, sodium nitrate, potassium sulfate and potassium nitrate to an electromembrane process for converting the least one of lithium sulfate, lithium nitrate, sodium sulfate, sodium nitrate, potassium sulfate, potassium nitrate, into at least one of least one of lithium hydroxide, sodium hydroxide and potassium hydroxide;

converting the at least one of least one of lithium hydroxide, sodium hydroxide, potassium hydroxide into at least one of at least one of least one of lithium carbonate, sodium carbonate and potassium hydroxide by a carbonatation process; and

reusing the at least one of lithium carbonate, sodium carbonate and potassium hydroxide obtained by the carbonatation process for reacting with the metal sulfate and/or metal nitrate.

[0020] According to another aspect, there is provided a process for preparing a metal oxide comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum, the process comprising:

reacting a metal sulfate and/or a metal nitrate comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum with lithium carbonate, sodium carbonate and/or potassium carbonate and optionally a chelating agent to obtain a solid comprising a metal carbonate comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum, and a liquid comprising at least one of lithium sulfate, lithium nitrate, sodium sulfate, sodium nitrate, potassium sulfate and potassium nitrate;

separating the liquid and the solid from one another to obtain the metal carbonate;

submitting the liquid comprising at least one of lithium sulfate, lithium nitrate, sodium sulfate, sodium nitrate, potassium sulfate and potassium nitrate to an electromembrane process for converting the at least one of lithium sulfate, lithium nitrate, sodium sulfate, sodium nitrate, potassium sulfate and potassium nitrate into at least one of least one of lithium hydroxide, sodium hydroxide and potassium hydroxide; and converting the at least one of least one of lithium hydroxide, sodium hydroxide, potassium hydroxide into at least one of at least one of least one of lithium carbonate, sodium carbonate and potassium carbonate by a carbonatation process; and

reusing at least a first portion of the at least one of lithium carbonate, sodium carbonate and potassium carbonate obtained by the carbonatation process for reacting with the metal sulfate and/or the metal nitrate;

reacting at least a second portion of the at least one of lithium carbonate, sodium carbonate and potassium carbonate obtained by the carbonatation process with the obtained metal carbonate to obtain a mixture of metal carbonates; and

roasting the mixture of metal carbonates to obtain the metal oxide.

[0021] According to another aspect of the present disclosure, there is provided a process for preparing a metal carbonate comprising at least one metal chosen from nickel, cobalt, manganese, lithium and aluminum, the process comprising:

reacting a first metal sulfate and/or a first metal nitrate comprising at least one metal chosen from nickel, cobalt, manganese, lithium and aluminum with a base comprising a second metal and optionally a chelating agent in order to obtain a solid comprising the metal carbonate and a liquid comprising at least one of a second metal sulfate and a second metal nitrate;

separating the liquid and the solid from one another to obtain the metal carbonate;

submitting the liquid comprising at least one of the second metal sulfate and the second metal nitrate to an electromembrane process for converting at least one of the second metal sulfate and the second metal nitrate into a second metal hydroxide;

converting the second metal hydroxide into a second metal carbonate that is at least one lithium carbonate, sodium carbonate and potassium carbonate by a carbonatation process; and reusing the second metal carbonate obtained by the carbonatation process for reacting with the first metal sulfate and/or the first metal nitrate.

[0022] According to another aspect, there is provided a process for preparing a metal oxide comprising at least one metal chosen from nickel, cobalt, manganese, lithium and aluminum, the process comprising:

reacting a first metal sulfate and/or a first metal nitrate comprising at least one metal chosen from nickel, cobalt, manganese, lithium and aluminum with a base comprising a second metal and optionally a chelating agent to obtain a solid comprising a metal carbonate comprising at least one metal chosen from nickel, cobalt, manganese, lithium and aluminum, and a liquid comprising at least one of a second metal sulfate and a second metal nitrate;

separating the liquid and the solid from one another to obtain the metal carbonate;

submitting the liquid comprising the at least one of a second metal sulfate and a second metal nitrate to an electromembrane process for converting the at least one of a second metal sulfate and a second metal nitrate into a second metal hydroxide;

converting the second metal hydroxide into a second metal carbonate that is at least one of lithium carbonate, sodium carbonate and potassium carbonate by a carbonatation process; and

reusing at least a first portion of the second metal carbonate obtained by the carbonatation process for reacting with the first metal sulfate and/or the first metal nitrate;

reacting at least a second portion of the second metal carbonate obtained by the carbonatation process with the obtained metal hydroxide to obtain a mixture of metal carbonates; and

roasting the mixture of metal carbonates to obtain the metal oxide.

[0023] According to another aspect, there is provided a process for preparing a metal hydroxide comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum, said process comprising:

reacting a metal sulfate comprising (i) lithium; (ii) at least one metal chosen from nickel and cobalt and optionally (iii) at least one metal chosen from manganese and aluminum with sodium hydroxide and optionally a chelating agent in order to obtain a solid comprising said metal hydroxide and a liquid comprising sodium sulfate and lithium sulfate;

separating said liquid and said solid from one another to obtain said metal hydroxide;

submitting said liquid comprising sodium sulfate and lithium sulfate to an electromembrane process for converting said sodium sulfate and said lithium sulfate into sodium hydroxide and lithium hydroxide; and

reusing said sodium hydroxide obtained by said electromembrane process for reacting with said metal sulfate.

[0024] According to another aspect, there is provided a process for preparing a metal hydroxide comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum, said process comprising:

reacting a metal sulfate comprising (i) lithium; (ii) at least one metal chosen from nickel and cobalt and optionally (iii) at least one metal chosen from manganese and aluminum with sodium hydroxide and optionally a chelating agent in order to obtain a solid comprising said metal hydroxide and a liquid comprising sodium sulfate and lithium sulfate;

separating said liquid and said solid from one another to obtain said metal hydroxide;

separating sodium sulfate and lithium sulfate from one another;

submitting said liquid comprising sodium sulfate to an electromembrane process for converting said sodium sulfate into sodium hydroxide; and

reusing said sodium hydroxide obtained by said electromembrane process for reacting with said metal sulfate.

[0025] According to another aspect, there is provided a process for preparing a metal hydroxide comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum, said process comprising:

reacting a metal sulfate comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum with sodium hydroxide and optionally a chelating agent in order to obtain a solid comprising said metal hydroxide and a liquid comprising sodium sulfate;

separating said liquid and said solid from one another to obtain said metal hydroxide;

submitting said liquid comprising sodium sulfate to an electromembrane process for converting said sodium sulfate into sodium hydroxide; and

reusing said sodium hydroxide obtained by said electromembrane process for reacting with said metal sulfate.

[0026] According to another aspect there is provided a process a process for preparing a metal carbonate comprising at least one metal chosen from nickel, cobalt, manganese, lithium and aluminum, the process comprising:

reacting a metal sulfate and/or a metal nitrate comprising at least one metal chosen from nickel, cobalt, manganese, lithium and aluminum with a base chosen from Li_2CO_3, Na_2CO_3, K_2CO_3, Rb_2CO_3, Cs_2CO_3, $MgCO_3$, $CaCO_3$, $SrCO_3$ and $BaCO_3$ and optionally a chelating agent in order to obtain a solid comprising the metal carbonate and a liquid comprising at least one of Li_2SO_4 Na_2SO_4, K_2SO_4, Rb_2SO_4, Cs_2SO_4, $MgSO_4$, $CaSO_4$, $SrSO_4$, $BaSO_4$, $LiNO_3$ $NaNO_3$, KNO_3, $RbNO_3$, $CsNO_3$, $Mg(NO_3)_2$, $Ca(NO_3)_2$, $Sr(NO_3)_2$ and $Ba(NO_3)_2$,

separating the liquid and the solid from one another to obtain the metal hydroxide;

submitting the liquid comprising at least one of Li_2SO_4 Na_2SO_4, K_2SO_4, Rb_2SO_4, Cs_2SO_4, $MgSO_4$, $CaSO_4$, $SrSO_4$, $BaSO_4$, $LiNO_3$ $NaNO_3$, KNO_3, $RbNO_3$, $CsNO_3$, $Mg(NO_3)_2$, $Ca(NO_3)_2$, $Sr(NO_3)_2$ and $Ba(NO_3)_2$ to an electromembrane process for converting the least one of Li_2SO_4 Na_2SO_4, K_2SO_4, Rb_2SO_4, Cs_2SO_4, $MgSO_4$, $CaSO_4$, $SrSO_4$, $BaSO_4$, $LiNO_3$ $NaNO_3$, KNO_3, $RbNO_3$,

$CsNO_3$, $Mg(NO_3)_2$, $Ca(NO_3)_2$, $Sr(NO_3)_2$ and $Ba(NO_3)_2$ into at least one of LiOH, NaOH, KOH, RbOH, CsOH, $Mg(OH)_2$, $Ca(OH)_2$, $Sr(OH)_2$, and $Ba(OH)_2$;

converting the at least one of LiOH, NaOH, KOH, RbOH, CsOH, $Mg(OH)_2$, $Ca(OH)_2$, $Sr(OH)_2$, and $Ba(OH)_2$ into Li_2CO_3, Na_2CO_3, K_2CO_3, Rb_2CO_3, Cs_2CO_3, $MgCO_3$, $CaCO_3$, $SrCO_3$ and $BaCO_3$ by a carbonatation process; and

reusing the at least one of Li_2CO_3, Na_2CO_3, K_2CO_3, Rb_2CO_3, Cs_2CO_3, $MgCO_3$, $CaCO_3$, $SrCO_3$ and $BaCO_3$ obtained by the carbonatation process for reacting with the metal sulfate and/or the metal nitrate.

[0027] According to another aspect, there is provided a process for preparing a metal oxide comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum, said process comprising:

reacting a metal sulfate and/or a metal nitrate comprising at least one metal chosen from nickel, cobalt, manganese, lithium and aluminum with a base chosen from Li_2CO_3, Na_2CO_3, K_2CO_3, Rb_2CO_3, Cs_2CO_3, $MgCO_3$, $CaCO_3$, $SrCO_3$ and $BaCO_3$ and optionally a chelating agent in order to obtain a solid comprising the metal carbonate and a liquid comprising at least one of Li_2SO_4 Na_2SO_4, K_2SO_4, Rb_2SO_4, Cs_2SO_4, $MgSO_4$, $CaSO_4$, $SrSO_4$, $BaSO_4$, $LiNO_3$ $NaNO_3$, KNO_3, $RbNO_3$, $CsNO_3$, $Mg(NO_3)_2$, $Ca(NO_3)_2$, $Sr(NO_3)_2$ and $Ba(NO_3)_2$,

separating said liquid and said solid from one another to obtain said metal carbonate;

submitting said liquid comprising at least one of Li_2SO_4 Na_2SO_4, K_2SO_4, Rb_2SO_4, Cs_2SO_4, $MgSO_4$, $CaSO_4$, $SrSO_4$, $BaSO_4$, $LiNO_3$ $NaNO_3$, KNO_3, $RbNO_3$, $CsNO_3$, $Mg(NO_3)_2$, $Ca(NO_3)_2$, $Sr(NO_3)_2$ and $Ba(NO_3)_2$ to an electromembrane process for converting at least one of Li_2SO_4 Na_2SO_4, K_2SO_4, Rb_2SO_4, Cs_2SO_4, $MgSO_4$, $CaSO_4$, $SrSO_4$, $BaSO_4$, $LiNO_3$ $NaNO_3$, KNO_3, $RbNO_3$, $CsNO_3$, $Mg(NO_3)_2$, $Ca(NO_3)_2$, $Sr(NO_3)_2$ and $Ba(NO_3)_2$ into at least one of LiOH, NaOH, KOH, RbOH, CsOH, $Mg(OH)_2$, $Ca(OH)_2$, $Sr(OH)_2$, or $Ba(OH)_2$;

converting the at least one of LiOH, NaOH, KOH, RbOH, CsOH, $Mg(OH)_2$, $Ca(OH)_2$, $Sr(OH)_2$, and $Ba(OH)_2$ into Li_2CO_3, Na_2CO_3, K_2CO_3,

Rb_2CO_3, Cs_2CO_3, $MgCO_3$, $CaCO_3$, $SrCO_3$ and $BaCO_3$ by a carbonatation process; and

reusing at least a first portion of said at least one of Li_2CO_3, Na_2CO_3, K_2CO_3, Rb_2CO_3, Cs_2CO_3, $MgCO_3$, $CaCO_3$, $SrCO_3$ and $BaCO_3$ obtained by said carbonatation process for reacting with said metal sulfate;

reacting at least a second portion of said at least one of Li_2CO_3, Na_2CO_3, K_2CO_3, Rb_2CO_3, Cs_2CO_3, $MgCO_3$, $CaCO_3$, $SrCO_3$ and $BaCO_3$ obtained by said carbonatation process with said obtained metal carbonate to obtain a mixture of metal carbonates ; and

roasting said mixture of metal carbonates to obtain said metal oxide.

[0028] According to another aspect, there is provided a process for preparing a metal oxide comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum, said process comprising:

reacting a metal sulfate comprising (i) lithium; (ii) at least one metal chosen from nickel and cobalt and optionally (iii) at least one metal chosen from manganese and aluminum with sodium hydroxide and optionally a chelating agent in order to obtain a solid comprising said metal hydroxide and a liquid comprising sodium sulfate and lithium sulfate;

separating said liquid and said solid from one another to obtain said metal hydroxide;

submitting said liquid comprising sodium sulfate and lithium sulfate to an electromembrane process for converting said sodium sulfate and said lithium sulfate into sodium hydroxide and lithium hydroxide;

separating said lithium hydroxide and said sodium hydroxide from one another;

reusing at least a first portion of said sodium hydroxide obtained by said electromembrane process for reacting with said metal sulfate;

reacting at least a first portion of said lithium hydroxide obtained by said electromembrane process with said obtained metal hydroxide to obtain a mixture of metal hydroxides; and

roasting said mixture of metal hydroxides to obtain said metal oxide.

[0029] According to another aspect, there is provided a process for preparing a metal oxide comprising at least one metal chosen from nickel, cobalt, manganese, lithium and aluminum, the process comprising:

reacting a first metal sulfate and/or a first metal nitrate comprising at least one metal chosen from nickel, cobalt, manganese, lithium and aluminum with a base comprising a second metal and optionally a chelating agent to obtain a solid comprising a metal hydroxide comprising at least one metal chosen from nickel, cobalt, manganese, lithium and aluminum, and a liquid comprising at least one of a second metal sulfate and a second metal nitrate;

separating the liquid and the solid from one another to obtain the metal hydroxide;

submitting the liquid comprising the at least one of a second metal sulfate and a second metal nitrate to an electromembrane process for converting the at least one of a second metal sulfate and a second metal nitrate into a second metal hydroxide; and

reusing at least a first portion of the second metal hydroxide obtained by the electromembrane process for reacting with the first metal sulfate and/or the first metal nitrate;

mixing a third metal hydroxide with the obtained metal hydroxide to obtain a mixture of metal hydroxides; and

roasting the mixture of metal hydroxides to obtain the metal oxide.

[0030] According to another aspect of the present disclosure, there is provided a process for preparing a metal hydroxide comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum, the process comprising:

reacting a metal sulfate comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum with sodium hydroxide and optionally a chelating agent in order to obtain a solid comprising the metal hydroxide and a liquid comprising sodium sulfate;

separating the liquid and the solid from one another to obtain the metal hydroxide;

submitting the liquid comprising sodium sulfate to an electromembrane process for converting the sodium sulfate into sodium hydroxide; and

reusing the sodium hydroxide obtained by the electromembrane process for reacting with the metal sulfate.

[0031] According to another aspect, there is provided a process for preparing a metal oxide comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum, the process comprising:

reacting a metal sulfate comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum with sodium hydroxide and optionally a chelating agent to obtain a solid comprising a metal hydroxide comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum, and a liquid comprising lithium sulfate;

separating the liquid and the solid from one another to obtain the metal hydroxide;

submitting the liquid comprising sodium sulfate to an electromembrane process for converting the sodium sulfate into sodium hydroxide; and

reusing at least a first portion of the sodium hydroxide obtained by the electromembrane process for reacting with the metal sulfate;

mixing another metal hydroxide with the obtained metal hydroxide to obtain a mixture of metal hydroxides; and

roasting the mixture of metal hydroxides to obtain the metal oxide.

[0032] According to another aspect, there is provided a process for preparing a metal hydroxide comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum, said process comprising:

reacting a metal sulfate comprising (i) at least one metal chosen from nickel and cobalt and optionally (iii) at least one metal chosen from manganese and aluminum with sodium hydroxide and optionally a chelating agent in order to obtain a solid comprising said metal hydroxide and a liquid comprising sodium sulfate and optionally lithium sulfate;

separating said liquid and said solid from one another to obtain said metal hydroxide;

submitting said liquid comprising sodium sulfate and optionally lithium sulfate to an electromembrane process for converting said sodium sulfate and optionally said lithium sulfate into sodium hydroxide and optionally lithium hydroxide; and

reusing said sodium hydroxide obtained by said electromembrane process for reacting with said metal sulfate.

[0033] According to another aspect, there is provided a process for preparing a metal hydroxide comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum, the process comprising:

reacting a metal sulfate comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum with lithium hydroxide, sodium hydroxide and/or potassium hydroxide and optionally a chelating agent in order to obtain a solid comprising the metal hydroxide and a liquid comprising lithium sulfate, sodium sulfate and/or potassium sulfate;

separating the liquid and the solid from one another to obtain the metal hydroxide;

submitting the liquid comprising lithium sulfate, sodium sulfate and/or potassium sulfate to an electromembrane process for converting the lithium sulfate, sodium sulfate and/or potassium sulfate into lithium hydroxide, sodium hydroxide and/or potassium hydroxide respectively;

reusing the sodium hydroxide obtained by the electromembrane process for reacting with the metal sulfate; and

reusing the lithium hydroxide obtained by the electromembrane process for reacting with the metal sulfate and/or with the metal hydroxide.

[0034] According to another aspect, there is provided the use of the metal hydroxide, the metal carbonate and/or the metal oxide obtained from a process described in the present disclosure in the manufacture of a cathode.

[0035] According to another aspect, there is provided a method of using the metal hydroxide, the metal carbonate and/or the metal oxide obtained from a process described in the present disclosure, the method comprising incorporating the metal hydroxide, the metal carbonate and/or the metal oxide in the manufacture of a cathode.

BRIEF DESCRIPTION OF DRAWINGS

[0036] In the following drawings, which represent by way of example only, various embodiments of the disclosure :

[0037] FIG. 1 is a schematic diagram of a process according to an embodiment of the present disclosure;

[0038] FIG. 2 is a X-Ray diffraction pattern of cobalt hydroxide $Co(OH)_2$ (in black) obtained using LiOH as base source and the theoretical diffraction peaks of this compound (vertical bars);

[0039] FIG. 3 is X-Ray diffraction pattern of cobalt hydroxide $Co(OH)_2$ (in black) obtained using NaOH as base source and the theoretical diffraction peaks of this compound (vertical bars);

[0040] FIG. 4 is a X-Ray diffraction pattern of $LiCoO_2$ (in black) obtained by using the Co(OH)2 of FIG. 2 (in black) and the theoretical diffraction peaks of this compound (vertical bars);

[0041] FIG. 5 is a X-Ray diffraction pattern of $LiCoO_2$ (in black) obtained by using the Co(OH)2 of FIG. 3 and the theoretical diffraction peaks of this compound (vertical bars);

[0042] FIG. 6 represent charge/discharge curves of $LiCoO_2$;

[0043] FIG. 7 is a X-Ray diffraction pattern of Nickel-Cobalt-Aluminum hydroxide $Ni_{0.8}Co_{0.15}Al_{0.05}O_2(OH)_2$ (in black) and the theoretical diffraction peaks of this compound (vertical bars);

[0044] FIG. 8 is a X-Ray diffraction pattern of the lithiated Nickel-Cobalt-Aluminum oxide $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$ (in black) and the theoretical diffraction peaks of this compound (vertical bars);

[0045] FIG. 9 is a charge/discharge curves of $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$;

[0046] FIG. 10 is a X-Ray diffraction pattern of Nickel-Manganese-Cobalt hydroxide $Ni_{0.8}Mn_{0.1}Co_{0.1}(OH)_2$ (in black) and the theoretical diffraction peaks of this compound (vertical bars);

[0047] FIG. 11 is X-Ray diffraction pattern of lithiated Nickel-Manganese-Cobalt oxide $LiNi_{0.8}Mn_{0.1}Co_{0.1}O_2$ (in black) and the theoretical diffraction peaks of this compound (vertical bars);

[0048] FIG. 12 is a X-Ray diffraction pattern of Nickel-Manganese-Cobalt hydroxide $Ni_{0.6}Mn_{0.2}Co_{0.2}(OH)_2$ (in black) and the theoretical diffraction peaks of this compound (vertical bars);

[0049] FIG. 13 : X-Ray diffraction pattern of lithiated Nickel-Manganese-Cobalt oxide $LiNi_{0.6}Mn_{0.2}Co_{0.2}O_2$ (in black) and the theoretical diffraction peaks of this compound (vertical bars);

[0050] FIG. 14 represent charge/discharge curves of $LiNi_{0.6}Mn_{0.2}Co_{0.2}O_2$;

[0051] FIG. 15 is a plot showing concentration of H_2SO_4 in the anolyte of a two-compartment cell as a function of time in an example of electrolysis of Li_2SO_4;

[0052] FIG. 16 is a plot showing conductivity of anolyte and catholyte in a two-compartment cell as a function of time in an example of electrolysis of Li_2SO_4;

[0053] FIG. 17 is a plot showing temperature of anolyte and catholyte in a two-compartment cell as a function of time in an example of electrolysis of Li_2SO_4;

[0054] FIG. 18 is a plot showing voltage in a two-compartment cell as a function of time in an example of electrolysis of Li_2SO_4;

[0055] FIG. 19 is a plot showing flow rate of $LiOH \cdot H_2O$ as a function of concentration of H_2SO_4 in a two-compartment cell in an example of electrolysis of Li_2SO_4;

[0056] FIG. 20 is another plot showing flow rate of $LiOH \cdot H_2O$ as a function of concentration of H_2SO_4 in a two-compartment cell in an example of electrolysis of Li_2SO_4;

[0057] FIG. 21 is a plot showing current efficiency as a function of concentration of H_2SO_4 in a two-compartment cell in an example of electrolysis of Li_2SO_4;

[0058] FIG. 22 is a plot showing productivity of $LiOH \cdot H_2O$ as a function of concentration of H_2SO_4 in a two-compartment cell in an example of electrolysis of Li_2SO_4;

[0059] FIG. 23 is a plot showing energy consumption as a function of concentration of H_2SO_4 in a two-compartment cell in an example of electrolysis of Li_2SO_4;

[0060] FIG. 24 is a schematic diagram of a process according to an embodiment of the present disclosure using LiOH as pH enhancer;

[0061] FIG. 25 is a schematic diagram of a process according to an embodiment of the present disclosure using NaOH as pH enhancer;

[0062] FIG. 26 is a schematic diagram of a process according to an embodiment of the present disclosure using LiOH and/or NaOH as pH enhancer;

[0063] FIG. 27 is a schematic diagram of a process according to an embodiment of the present disclosure using NaOH as pH enhancer for a metalu sulfate solution containing Lithium ions;

[0064] FIG. 28 is a schematic diagram of a process according to an embodiment of the present disclosure, with purification and/or concentration of the sulfate solution recovered before electromembrane process;

[0065] FIG. 29 is a schematic diagram of a process according to an embodiment of the present disclosure, with purification and/or concentration of the sulfate solution recovered before electromembrane process and concentration of the anolyte solution, and addition of H_2O_2;

[0066] FIG. 30 is a schematic diagram of a process according to an embodiment of the present disclosure for the core-shell synthesis using LiOH and/or NaOH as pH enhancer;

[0067] FIG. 31 is a schematic diagram of a process according to an embodiment of the present disclosure for the synthesis of a lithiated metal oxide using Li2CO3 as pH enhancer for the precipitation of metal carbonate.

[0068] FIG. 32 is a schematic diagram of a process according to an embodiment of the present disclosure using nitric acid for the leaching of the transition metal source.

[0069] FIG. 33 is a schematic diagram of a process for the production of high purity sulfate salts using H_2SO_4 to leach the nickel cobalt concentrate;

[0070] FIG. 34 is a schematic diagram of a process for the production of high purity sulfate salts; and

[0071] FIG. 35 is a schematic diagram of a process for the production of high purity sulfate salts.

DETAILLED DESCRIPTION OF VARIOUS EMBODIMENTS

[0072] Unless otherwise indicated, the definitions and embodiments described in this and other sections are intended to be applicable to all embodiments and aspects of the present disclosure herein described for which they are suitable as would be understood by a person skilled in the art.

[0073] As used in the present disclosure, the singular forms “a”, “an” and “the” include plural references unless the content clearly dictates otherwise.

[0074] In understanding the scope of the present disclosure, the term “comprising” and its derivatives, as used herein, are intended to be open ended terms that specify the presence of the stated features, elements, components, groups, integers, and/or steps, but do not exclude the presence of other unstated features, elements, components, groups, integers and/or steps. The foregoing also applies to words having similar meanings such as the terms, “including”, “having” and their derivatives. The term “consisting” and its derivatives, as used herein, are intended to be closed terms that specify the presence of the stated features, elements, components, groups, integers, and/or steps, but exclude the presence of other unstated features, elements, components, groups, integers and/or steps. The term “consisting essentially of”, as used herein, is intended to specify the presence of the stated features, elements, components, groups, integers, and/or steps as well as those that do not materially affect the basic and novel characteristic(s) of features, elements, components, groups, integers, and/or steps.

[0075] Terms of degree such as “about” and “approximately” as used herein mean a reasonable amount of deviation of the modified term such that the end result is not significantly changed. These terms of degree should be construed as including a deviation of ±10% of the modified term if this deviation would not negate the meaning of the word it modifies.

[0076] The term “suitable” as used herein means that the selection of the particular conditions would depend on the specific manipulation or operation to be performed, but the selection would be well within the skill of a person trained in the art. All processes described herein are to be conducted under conditions sufficient to provide the desired product. A person skilled in the art would understand that all reaction conditions, including, when applicable, for example, reaction time, reaction temperature, reaction pressure, reactant ratio, flow rate, reactant purity, current density, voltage, concentration, pH, oxidation reduction potential, cell area, type of membrane used, and recycle rates can be varied to optimize the yield of the desired product and it is within their skill to do so.

[0077] The expression “is at least substantially maintained” as used herein when referring to a value of a pH or a pH range that is maintained during a process of the disclosure or a portion thereof (for example, electrolysis, etc.) refers to maintaining the value of the pH or the pH range at least 75, 80, 85, 90, 95, 96, 97, 98 or 99 % of the time during the process or the portion thereof.

[0078] The expression “is at least substantially maintained” as used herein when referring to a value of a concentration or a concentration range that is maintained during a process of the disclosure or a portion thereof (for example, electrolysis, etc.) refers to maintaining the value of the concentration or the concentration range at least 75, 80, 85, 90, 95, 96, 97, 98 or 99 % of the time during the process or the portion thereof.

[0079] The expression “is at least substantially maintained” as used herein when referring to a value of a temperature or a temperature range that is maintained during a process of the disclosure or a portion thereof (for example, electrolysis, etc.) refers to maintaining the value of the temperature or the temperature range at least 75, 80, 85, 90, 95, 96, 97, 98 or 99 % of the time during the process or the portion thereof.

[0080] The expression “is at least substantially maintained” as used herein when referring to a value of an electrical current density or an electrical current density range that is maintained during a process of the disclosure or a portion thereof (for example, electrolysis, etc.) refers to maintaining the value of the electrical current density or the electrical current density range at least 75, 80, 85, 90, 95, 96, 97, 98 or 99 % of the time during the process or the portion thereof.

[0081] The expression “is at least substantially maintained” as used herein when referring to a value of an electrical current efficiency or an electrical current efficiency range that is maintained during a process of the disclosure or a portion thereof (for example, electrolysis, etc.) refers to maintaining the value of the electrical current efficiency or the electrical current efficiency range at least 75, 80, 85, 90, 95, 96, 97, 98 or 99 % of the time during the process or the portion thereof.

[0082] The expression “is at least substantially maintained” as used herein when referring to a value of a voltage or a voltage range that is maintained during a process of the disclosure or a portion thereof (for example, electrolysis, etc.) refers to maintaining the value of the voltage or the voltage range at least 75, 80, 85, 90, 95, 96, 97, 98 or 99 % of the time during the process or the portion thereof.

[0083] The term “electromembrane process” as used herein refers, for example to a process that uses ion-exchange membrane(s) and an electric potential difference as the driving force for ionic species. The electromembrane process can be, for example (a membrane) electrodialysis or (a membrane) electrolysis. For example, the electromembrane process can be a membrane electrolysis.

[0084] The term “carbonatation process” as used herein refers, for example to a process in which a metal hydroxide will be converted into a metal carbonate. For example, such a process can involve the use of gaseous CO_2. For example, such a process can involve bubbling of CO_2.

[0085] The below presented examples are non-limitative and are used to better exemplify the processes of the present disclosure.

[0086] For example, the hydroxide can be chosen from nickel-cobalt-manganese hydroxides, nickel-cobalt-aluminum hydroxides, lithium-cobalt hydroxides, nickel hydroxides, nickel-cobalt-manganese oxyhydroxides, nickel-cobalt-aluminum oxyhydroxides, nickel oxyhydroxides and lithium-cobalt oxyhydroxides.

[0087] For example, the oxide can be chosen from nickel-cobalt-manganese oxides, nickel-cobalt-aluminum oxides, nickel oxide, lithiumnickel-cobalt-manganese oxides, lithium nickel-cobalt-aluminum oxides, lithium nickel oxide and lithium-cobalt oxides.

[0088] For example, the solid is a precipitate comprising the metal hydroxide, the precipitate being obtained at a pH of about 8 to about 14.

[0089] For example, the solid is a precipitate comprising the metal hydroxide, the precipitate being obtained at a pH of about 9 to about 13.

[0090] For example, the solid is a precipitate comprising the metal hydroxide, the precipitate being obtained at a pH of about 10 to about 12.

[0091] For example, the process further comprises washing the metal hydroxide.

[0092] For example, the process further comprises drying the metal hydroxide at a temperature of about 80°C to about 130°C or 90°C to about 120°C.

[0093] For example, the metal sulfate is reacted with lithium hydroxide and a chelating agent that is ammonia.

[0094] For example, the metal sulfate is reacted with lithium carbonate and a chelating agent that is ammonia.

[0095] For example, the metal sulfate is reacted with lithium carbonate and a chelating agent that is ammonia hydrogen carbonate.

[0096] For example, the first metal can be chosen from nickel, cobalt, manganese, lithium and aluminum.

[0097] For example, the base can comprise at least one of LiOH, NaOH, KOH, RbOH, CsOH, $Mg(OH)_2$, $Ca(OH)_2$, $Sr(OH)_2$ and $Ba(OH)_2$.

[0098] For example, the base can comprise at least one of be Li_2CO_3, Na_2CO_3, K_2CO_3, Rb_2CO_3, Cs_2CO_3, $MgCO_3$, $CaCO_3$, $SrCO_3$ and $BaCO_3$.

[0099] For example, the base can comprise at least one of $LiHCO_3$, $NaHCO_3$, $KHCO_3$, $RbHCO_3$, $CsHCO_3$, $Mg(HCO_3)_2$, $Ca(HCO_3)_2$, $Sr(HCO_3)_2$ and $Ba(HCO_3)_2$.

[00100] For example, the metal hydroxide can comprise at least one of LiOH, NaOH, KOH, RbOH, CsOH, $Mg(OH)_2$, $Ca(OH)_2$, $Sr(OH)_2$ and $Ba(OH)_2$.

[00101] For example, the second metal can be Li, Na, K, Rb, Cs, Mg, Ca, Sr, or Ba.

[00102] For example, the third metal can be Li, Na, Ni, Co, Mn, Al, K, Rb, Cs, Mg, Ca, Sr, or Ba.

[00103] For example, the third metal hydroxide can be LiOH.

[00104] For example, the another metal can be Li, Na, Ni, Co, Mn, Al, K, Rb, Cs, Mg, Ca, Sr, or Ba.

[00105] For example, the another metal hydroxide can be LiOH.

[00106] For example, the base can be purified before being reacted with the metal sulfate. For example, the base can be crystallized.

[00107] For example, the metal hydroxide produced by the electromembrane process can be purified before being reacted with the metal sulfate. For example, the metal hydroxide can be crystallized.

[00108] For example, before submitting the liquid comprising sulfate to an electromembrane process in order to obtain an hydroxide, the sulfate can be purified and/or concentrated.

[00109] For example, the chelating agent can be chosen from NH_3, NH_4OH, acetylacetone, 5-sulfosalicylic acid, oxalic acid.

[00110] For example, the chelating agent can be chosen from EDTA (ethylenediaminetetraacetic acid) NTA (nitrilotriacetic acid), DCTA (trans-1,2-diaminocyclohexanetetraacetic acid), DTPA (diethylene-triamine pentaacetic acid), and EGTA (ethylene glycol bis(2-aminoethyl ether)-N,N,N',N'-tetraacetic acid).

[00111] For example, the chelating agent can be present.

[00112] For example, if the electromembrane process is a Na-based process, a purification step for the separation of lithium (in solution as lithium sulfate) from the sodium sulfate solution can be carried out.

[00113] For example, sodium sulfate and lithium sulfate can be separated from one another.

[00114] For example, sodium sulfate and lithium sulfate can be separated from one another by means of a crystallization.

[00115] For example, the metal hydroxide can be $NiCoAl(OH)_2$ or $NiMnCo(OH)_2$.

[00116] For example, the metal hydroxide can be chosen from $Ni_{0.8}Co_{0.15}Al_{0.05}(OH)_2$, $Ni_{0.8}Mn_{0.1}Co_{0.1}(OH)_2$ and $Ni_{0.6}Mn_{0.2}Co_{0.2}(OH)_2$.

[00117] For example, the metal oxide can be of formula $LiMO_2$, wherein M is at least one metal chosen from nickel, cobalt, manganese, lithium and aluminum.

[00118] For example, the metal oxide can be of formula LiM_2O_4, wherein M is at least one metal chosen from nickel, cobalt, manganese, lithium and aluminum.

[00119] For example , the metal hydroxide or metal oxide can be of core-shell type.

[00120] For example, the metal oxide can be chosen from $LiNi_{0.33}Mn_{0.33}Co_{0.33}O_2$, $LiNi_{0.5}Mn_{0.3}Co_{0.2}O_2$, $LiNi_{0.6}Mn_{0.2}Co_{0.2}O_2$, $LiNi_{0.8}Mn_{0.1}Co_{0.1}O_2$ and $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$, or $[LiNi_xM1_yM2_zO_2]_{core}/[LiNi_aM1_bM2_cO_2]_{shell}$, with M1 = Mn, Co or Al and M2 = Mn, Co or Al with $x+y+z = 1$, $a+b+c = 1$.

[00121] For example, the metal oxide can be of formula LiMO2, or $Li(1+x)M(1_{-x})O_2$ for lithium-rich and $Li_{(1-z)}M_{(1+z)}O_2$ for Li-deficient, wherein M can be at least one metal chosen from nickel, cobalt, manganese, lithium and aluminum.

[00122] For example, the lithium hydroxide obtained by the electromembrane process can be used as is in aqueous composition and reacted with the obtained metal hydroxide to obtain a mixture of metal hydroxides.

[00123] For example, the lithium hydroxide obtained by the electromembrane process can be crystallized before being reacted with the obtained metal hydroxide to obtain a mixture of metal hydroxides.

[00124] For example, the lithium hydroxide obtained by the electromembrane process can be crystallized and and then dissolved before being reacted with the obtained metal hydroxide to obtain a mixture of metal hydroxides.

[00125] For example, the roasting of the mixture of metal hydroxides comprises roasting at a first temperature of at least 350 °C for a period of time of at least about 4 hours.

[00126] For example, roasting the mixture of metal hydroxides comprises roasting at a first temperature of at least about 400 °C for a period of time of at least about 6 hours.

[00127] For example, the process can further comprise roasting the mixture of metal hydroxides comprises roasting at a second temperature of at least about 600 °C for a period of time of at least about 6 hours.

[00128] For example, the process can further comprise roasting the mixture of metal hydroxides comprises roasting at a second temperature of at least about 700 °C for a period of time of at least about 8 hours.

[00129] For example, the process can further comprise roasting the mixture of metal hydroxides comprises roasting at a second temperature of at least about 500 °C for a period of time of at least about 8 hours.

[00130] For example, NH_3 can be recovered *in situ* during mixture formation.

[00131] For example, the electromembrane process for converting Li_2SO_4 into LiOH can be chosen from electromembrane processes as described in any one of WO2013159194, WO2013177680, WO2014138933, WO 2015058287, WO2015058288, WO2015123762 and WO2017/031595.

[00132] For example, carbonatation can be carried out as described in WO2013177680 or in WO2015058287.

[00133] The processes of the present disclosure can be operated, for example as a batch process. Alternatively, the processes of the present disclosure can be operated as a semi-continuous process or a continuous process.

[00134] It will be appreciated by a person skilled in the art that one or more parameters of the processes of the present disclosure such as but not limited to pH, temperature, current density, voltage, current efficiency and concentration can be monitored, for example by means known in the art. The selection of a suitable means for monitoring a particular parameter in a process of the present disclosure can be made by a person skilled in the art. Such parameters can also be maintained and/or changed by a person skilled in the art, for example in light of their common general knowledge and with reference to the present disclosure.

[00135] The person skilled in the art would understand that various different sources can be used for the metal sulfates. Metal sulfate(s) can be purchased. Metal sulfates can also be obtained by leaching a metal or a mixture of metals with H_2SO_4. Metal sulfate(s) can be obtained by leaching of spent lithium ion batteries. Metal sulfate(s) can be obtained by leaching a residue obtained after crushing spent lithium ion batteries. Metal sulfate(s) can be obtained by leaching a residue after treatment of spent lithium ion batteries Metal sulfate(s) can for example be derived from a mixture of transition metals that have been leached. Metal sulfate(s) can be provided from a concentrate derived from a mining company. Metal sulfate(s) can be obtained by leaching of a nickel ore containing cobalt.

[00136] For example, during the electromembrane process consumption of the lithium sulf ate to prepare lithium hydroxide can proceed to a pre-determined extent.

[00137] For example, the composition comprising lithium sulfate can also comprise H_2SO_4.

[00138] For example, in the processes of the present disclosure, the aqueous composition comprising the lithium sulfate is submitted to an electromembrane process under suitable conditions for conversion of the lithium sulfate to lithium hydroxide to proceed to a pre-determined extent. The selection of a suitable pre-determined extent for a particular process of the present disclosure can be made by a person skilled in the art. For example, the aqueous composition comprising lithium sulfate is submitted to a electromembrane process under suitable conditions for consumption of the lithium sulfate to prepare lithium hydroxide until one or more competing side reactions proceed to a pre-determined extent, for example to an extent such that the preparation of lithium hydroxide is no longer efficient.

[00139] For example, the electromembrane process is a two-compartment monopolar or bipolar membrane electrolysis process carried out in an electrochemical cell comprising an anolyte compartment separated from a catholyte compartment by a cation exchange membrane, conversion of the lithium sulfate to lithium hydroxide can proceed until hydroxide current efficiency is no longer efficient, for example hydroxide current efficiency is no longer at least substantially maintained so that it decreases. For example, the electromembrane process is a two-compartment monopolar or bipolar membrane electrolysis process carried out in a electrochemical cell comprising an anolyte compartment separated from a catholyte compartment by a cation exchange membrane, conversion of the lithium sulfate to lithium hydroxide can proceed until pH in the anolyte compartment is a value of about 0.3 to about 1.4, about 0.4 to about 1.2, about 0.4 to about 1.2, about 0.5 to about 0.8, about 0.5 to about 0.7 or about 0.6.

[00140] For example, the electromembrane process is a two-compartment monopolar or bipolar membrane electrolysis process carried out in a electrochemical cell comprising an anolyte compartment separated from a catholyte compartment by a cation exchange membrane, conversion of the lithium sulfate to lithium hydroxide can proceed until consumption of a particular amount of the lithium sulfate comprised within the aqueous composition.

[00141] For example, the pre-determined extent can comprise consumption of about 30 to about 60 weight % or of about 30 to about 50 weight % of the lithium sulfate comprised within the aqueous composition, based on the total amount of lithium sulfate contained in the aqueous composition. For example, the pre-determined extent can comprise consumption of about 35 to about 45 weight % of the lithium sulfate comprised within the aqueous composition.

[00142] For example, the electromembrane process can comprise, consist essentially of or consist of a three-compartment membrane electrolysis process, for example a three-compartment monopolar or bipolar membrane electrolysis process.

[00143] For example, the electromembrane process can comprise, consist essentially of or consist of a two-compartment membrane electrolysis process, for example a two-compartment monopolar or bipolar membrane electrolysis process.

[00144] For example, the electromembrane process can comprise, consist essentially of or consist of a three-compartment membrane electrolysis process, for example a three-compartment bipolar membrane electrolysis process.

[00145] For example, the electromembrane process can comprise, consist essentially of or consist of a two-compartment membrane electrolysis process, for example a two-compartment bipolar membrane electrolysis process.

[00146] For example, the two-compartment membrane electrolysis process such as the two-compartment monopolar or bipolar membrane electrolysis process can be carried out in a electrochemical cell comprising an anolyte compartment separated from a catholyte compartment by a cation exchange membrane.

[00147] For example, the cation exchange membrane can comprise, consist essentially of or consist of a perfluorosulfonic acid such as a Nafion^{TM} 324 (or perfluorinate sulfonic acid), a cation exchange membrane or other membranes used for caustic concentration such as FuMA-Tech FKB^{TM} or Astom CMB cation exchange membranes. The selection of a suitable cation exchange membrane for a particular process of the present disclosure can be made by a person skilled in the art.

[00148] For example, during the two-compartment membrane electrolysis process such as the two-compartment monopolar or bipolar membrane electrolysis process, an aqueous stream comprising the lithium sulfate can be introduced into the anolyte compartment, the first lithium-reduced aqueous stream can be removed from the anolyte compartment and the first lithium hydroxide-enriched aqueous stream can be removed from the catholyte compartment.

[00149] For example, in the catholyte compartment of the two-compartment monopolar or bipolar membrane electrolysis process, lithium hydroxide can be at least substantially maintained at a concentration of about 1 M to about 4 M, about 2 M to about 4 M, about 2 M to about 3 M, about 2.5 to about 3.5 M, about 2.8 to about 3.2 M or about 3 M.

[00150] For example, during the two-compartment monopolar or bipolar membrane electrolysis process, the aqueous stream comprising the lithium sulfate can be introduced into the anolyte compartment at a temperature of about 10 °C to about 100°C, about 10 °C to about 100°C, about 10 °C to about 90°C, about 20 °C to about 85°C, about 40 °C to about 80°C, about 40 °C to about 70°C, about 45 °C to about 60 °C, about 45 °C to about 55°C or about 50°C.

[00151] For example, during the two-compartment monopolar or bipolar membrane electrolysis process, the first lithium-reduced aqueous stream can be removed from the anolyte compartment at a temperature of about 20 °C to about 100 °C, about 20 °C to about 85 °C, about 50 °C to about 85 °C, about 55 °C to about 65 °C, about 45 °C to about 60 °C about 60 °C to about 85 °C, about 70 °C to about 85 °C or about 80°C.

[00152] For example, during the two-compartment monopolar or bipolar membrane electrolysis process, temperature in an electrochemical cell can be at least substantially maintained at a value of about 60°C to about 110°C, about 60°C to about 100°C, about 60°C to about 90°C, about 60°C to about 85°C, about 50°C to about 85°C, about 50°C to about 70°C, about 55°C to about 65°C, about 75°C to about 85°C or about 80°C.

[00153] For example, in the two-compartment monopolar or bipolar membrane electrolysis process, current density can be at least substantially maintained at a value of from about 0.1 kA/m^2 to about 8000 kA/m^2, 0.5 kA/m^2 to about 6 kA/m^2, about 1 kA/m^2 to about 6 kA/m^2, about 2 kA/m^2 to about 6 kA/m^2 or about 3 kA/m^2 to about 5 kA/m^2. For example, current density can be at least substantially maintained at a value chosen from about 3 kA/m^2, about 4 kA/m^2 and about 5 kA/m^2. For example, current density can be at least substantially maintained at a value of about 4 kA/m^2.

[00154] For example, in the two-compartment monopolar or bipolar membrane electrolysis process, voltage can be at least substantially maintained at a value of about 3 V to about 8 V, about 5 V to about 10 V, about 4 V to about 6 V, about 4 to about 5 or about 4.5.

[00155] For example, the electrochemical cell can have a surface area of about 0.2 m^2 to about 4 m^2, about 0.5 m^2 to about 3.5 m^2, about 1 m^2 to about 3 m^2 or about 1 m^2 to about 2 m^2.

[00156] For example, the electromembrane process can comprise, consist essentially of or consist of a two-compartment membrane electrolysis process, for example a two-compartment monopolar or bipolar membrane electrolysis process.

[00157] For example, the electromembrane process can comprise, consist essentially of or consist of a three-compartment membrane electrolysis process, for example a three-compartment monopolar or bipolar membrane electrolysis process.

[00158] For example, the three-compartment membrane electrolysis process such as the three-compartment monopolar or bipolar membrane electrolysis process can be carried out in a electrochemical cell comprising an anolyte compartment separated from a central compartment by an anion exchange membrane and a catholyte compartment separated from the central compartment by a cation exchange membrane.

[00159] For example, the cation exchange membrane can comprise, consist essentially of or consist of a perfluorsulfonic acid such as a Nafion^{TM} 324 cation exchange membrane or other membranes used for caustic concentration such as FuMA-Tech FKB or Astom CMB cation exchange membranes. The selection of a suitable cation exchange membrane for a particular process of the present disclosure can be made by a person skilled in the art.

[00160] For example, during the three-compartment membrane electrolysis process such as the three-compartment monopolar or bipolar membrane electrolysis process, the first lithium-reduced aqueous stream can be introduced into the central compartment, the second lithium-reduced aqueous stream can be removed from the central compartment and the second lithium hydroxide-enriched aqueous stream can be removed from the catholyte compartment.

[00161] For example, the three-compartment membrane electrolysis process such as the three-compartment monopolar or bipolar membrane electrolysis process can further comprise producing an acid such as sulfuric acid in the anolyte compartment and removing an acid-containing aqueous stream such as a sulfuric acid-containing aqueous stream from the anolyte compartment.

[00162] The selection of a suitable anion exchange membrane for a particular process of the present disclosure can be made by a person skilled in the art. For example, it will be appreciated by a person skilled in the art that a proton-blocking membrane may, for example be useful in processes coproducing acids such as sulfuric acid. For example, in the three-compartment monopolar or bipolar membrane electrolysis process, the anion exchange membrane can be a proton-blocking membrane. For example, the proton-blocking membrane can such as a Fumatech FAB, Astom ACM or Asahi AAV anion exchange membrane.

[00163] For example, in the anolyte compartment of the three-compartment monopolar or bipolar membrane electrolysis process, the acid such as sulfuric acid can be at least substantially maintained at a concentration of acid such as sulfuric acid of about 0.1 M to about 2 M. For example, in the anolyte compartment of the three-compartment monopolar or bipolar membrane electrolysis process, the sulfuric acid can be at least substantially maintained at a concentration of sulfuric acid can be about 0.5 M to about 1.5 M, about 0.7 M to about 1.2 M, or about 0.8 M.

[00164] For example, in the catholyte compartment of the three-compartment membrane electrolysis process, the lithium hydroxide can be at least substantially maintained at a concentration of about 1 M to about 5.0 M , about 1 M to about 4.0 M, about 1 M to about 3.0 M, about 2 M to about 3.0 M, about 1.5 M to about 2.5 M, about 1.8 M to about 2.2 M, or about 2 M.

[00165] For example, during the three-compartment monopolar or bipolar membrane electrolysis process, the first lithium-reduced aqueous stream can be introduced into the central compartment at a temperature of about 20 °C to about 85 °C, about 40 °C to about 85 °C, about 40 °C to about 75 °C, about 50 °C to about 70 °C, about 50 °C to about 65 °C or about 60°C.

[00166] For example, during the three-compartment monopolar or bipolar membrane electrolysis process, the second lithium-reduced aqueous stream can be removed from the anolyte compartment at a temperature of about 20 °C to about 80 °C, about 30 °C to about 70 °C, about 40 °C to about 80 °C or about 60°C.

[00167] For example, during the three-compartment monopolar or bipolar membrane electrolysis process, temperature in the second electrochemical cell can be at least substantially maintained at a value of about 30 °C to about 90°C, about 40 °C to about 85°C, about 50 °C to about 80°C, about 50 °C to about 70°C, about 50 °C to about 65°C, about 50°C to about 70°C, about 55°C to about 65°C, or about 60°C.

[00168] For example, in the three-compartment monopolar or bipolar membrane electrolysis process, current density can be at least substantially maintained at a value of about 0.5 kA/m^2 to about 5 kA/m^2, about 1 kA/m^2 to about 2 kA/m^2, about 3 kA/m^2 to about 5 kA/m^2, about 4 kA/m^2 or about 1.5 kA/m^2.

[00169] For example, in the three-compartment monopolar or bipolar membrane electrolysis process, voltage can be at least substantially maintained at a value of about 5 V to about 9 V, about 6 V to about 8 V , about 6.5 V to about 7.5 V or about 7 V.

[00170] For example, the electrochemical cell can have a cell area of about 0.2 m^2 to about 4 m^2, about 0.5 m^2 to about 3.5 m^2, about 1 m^2 to about 3 m^2 or about 1 m^2 to about 2 m^2.

[00171] Alternatively, for example, in the processes of the present disclosure, the three compartment monopolar or bipolar membrane electrolysis process can further comprise introducing ammonia into the anolyte compartment, producing an ammonium compound such as ammonium sulfate in the anolyte compartment and removing an ammonium compound-containing aqueous stream such as an ammonium sulfate-containing aqueous stream from the anolyte compartment.

[00172] The selection of a suitable anion exchange membrane for a particular process of the present disclosure can be made by a person skilled in the art. For example, it will be appreciated by a person skilled in the art that in processes that do not coproduce acids such as sulfuric acid, an anion exchange membrane that is not a proton-blocking membrane may be useful as it may, for example be able to withstand higher temperatures and/or have lower resistance than a proton-blocking membrane. For example, in the three-compartment monopolar or bipolar membrane electrolysis process, the anion exchange membrane may not be a proton-blocking membrane. For example, the anion exchange membrane can be a such as an Astom AHA anion exchange membrane or FuMA-Tech FAP.

[00173] For example, in the anolyte compartment of the three-compartment monopolar or bipolar membrane electrolysis process, the ammonium compound such as ammonium sulfate can be at least substantially maintained at a concentration of ammonium compound such as ammonium sulfate of about 0.5 M to about 5M, about 1 M to about 4M or about 3 M.

[00174] For example, in the catholyte compartment of the three-compartment monopolar or bipolar membrane electrolysis process, the lithium hydroxide can be at least substantially maintained at a concentration of about 1 M to about 4.0 M, about 1.5 M to about 2.5 M or about 2 M.

[00175] For example, pH in the anolyte compartment of the two-compartment monopolar or bipolar membrane electrolysis process and/or the central compartment of the three-compartment monopolar or bipolar membrane electrolysis process can be at least substantially maintained. For example, pH can be at least substantially maintained by adjusting at least one of current density of the two-compartment monopolar or bipolar membrane electrolysis process, current density of the three-compartment monopolar or bipolar membrane electrolysis process, flow rate of the first lithium-reduced aqueous stream and flow rate of the second lithium-reduced aqueous stream.

[00176] For example, during the two-compartment monopolar or bipolar membrane electrolysis process conversion of the lithium sulfate to lithium hydroxide can proceed to a pre-determined extent.

[00177] For example, during the two-compartment monopolar or bipolar membrane electrolysis process, an aqueous stream comprising the lithium sulfate can be introduced into the anolyte compartment, the first lithium-reduced aqueous stream can be removed from the anolyte compartment and the first lithium hydroxide-enriched aqueous stream can be removed from the catholyte compartment; and during the three-compartment monopolar or bipolar membrane electrolysis process, the first lithium-reduced aqueous stream can be introduced into the central compartment, the second lithium-reduced aqueous stream can be removed from the central compartment and the second lithium hydroxide-enriched aqueous stream can be removed from the catholyte compartment.

[00178] For example, the process can further comprise recycling at least a portion of the second lithium-reduced aqueous stream to the two-compartment monopolar or bipolar membrane electrolysis process.

[00179] It will be appreciated by a person skilled in the art that the process can also be varied, as appropriate, using the examples discussed herein.

[00180] For example, at least a portion of the processes of the present disclosure can be operated as a batch process. Alternatively, for example, the processes can be operated as a continuous process or a semi-continuous process. For example, it would be appreciated by a person skilled in the art that pH in the anolyte compartment of the two-compartment monopolar or bipolar membrane electrolysis process and/or the central compartment of the three-compartment monopolar or bipolar membrane electrolysis cell can be at least substantially maintained by adjusting the current density of the two-compartment monopolar or bipolar membrane electrolysis process and/or the three-compartment monopolar or bipolar membrane electrolysis process and/or the flow rate of the streams flowing between the processes, for example as described herein.

[00181] For example, pH in the anolyte compartment of the two-compartment monopolar or bipolar membrane electrolysis process and/or the central compartment of the three-compartment monopolar or bipolar membrane electrolysis process can be at least substantially maintained.

[00182] For example, pH can be at least substantially maintained by adjusting at least one of current density of the two-compartment monopolar or bipolar membrane electrolysis process, current density of the three-compartment monopolar or bipolar membrane electrolysis process, flow rate of the first lithium-reduced aqueous stream and flow rate of the second lithium-reduced aqueous stream.

[00183] The selection of a suitable means for measuring and/or monitoring pH can be made by a person skilled in the art. The selection of a suitable current density and/or a suitable flow rate can be made by a person skilled in the art.

[00184] For example, the process can further comprise removing a first hydrogen-containing stream from the catholyte compartment of the electrochemical cell. For example, the process can further comprise removing an oxygen-containing stream from the anolyte compartment of the electrochemical cell.

[00185] For example, the electrochemical cell can further comprise means to measure pH in the anolyte compartment, and the system is configured to convey the first lithium-reduced aqueous stream when pH in the anolyte compartment is below a pre-determined value.

[00186] For example, the electrochemical cell can further comprises means to measure pH in the central compartment, and the system is configured to convey unconverted lithium sulfate from the central compartment of the electrochemical cell when pH in the central compartment is above a pre-determined value.

[00187] For example, the electrochemical cell can further comprises means to measure concentration of lithium hydroxide in the catholyte compartment of the second electrochemical cell.

[00188] For example, lithium hydroxide can be crystallized as lithium hydroxide monohydrate, optionally dried and reacted in solid state with the obtained metal hydroxide to obtain a mixture of metal hydroxides.

[00189] For example, the metal sulfates can be obtained by leaching a battery.

[00190] For example, the battery can comprise LFP ($LiFePO_4$).

[00191] For example, lithium hydroxide can be concentrated before reacting it with the metal hydroxide and to form the mixture of metal hydroxides.

[00192] For example, concentration can be carried out by using reverse osmosis or by heating.

[00193] For example, lithium hydroxide can be crystallized before reacting it with the metal hydroxide and to form the mixture of metal hydroxides.

[00194] For example, the metal oxide can have the lamellar structure $Li(M^{2+})O_2$.

[00195] For example, the metal oxide can have the spinel structure $Li(M^{x+})_2O_4$, avec $3 < X < 4$.

[00196] For example, the lithium hydroxide composition can be concentrated before being reacted with the metal sulfate.

[00197] For example, concentration can be carried out by using reverse osmosis or by heating.

[00198] For example, the chelating agent can be NH_3.

[00199] For example, LiOH can be concentrated and then directly reacted with the metal hydroxide without crystallisation. For example, LiOH can be concentrated, crystallized, optionally dried and then directly reacted with the metal hydroxide.

[00200] For example, LiOH can be treated with a flash dryer.

[00201] For example LiOH and the metal hydroxide can be reacted together to obtain a mixture and then heated together.

[00202] For example LiOH and the metal hydroxide can be reacted together to obtain a mixture and then heated together in a spray dryer.

[00203] For example, crystals of lithium sulfate monohydrate can be inserted into the cell so as to increase concentration Li_2SO_4.

[00204] For example, the sulfate or hydroxide can be purified by a solvent extraction method. For example, the solvents used for solvent extraction can be based on phosphorous acid e.g. Cyanex™ 272, Cyanex™ 301, Cyanex™ 302, Di-(2-ethylhexyl)phosphoric acid (D2EHPA), DEHTPA, Baysolvex DEDP™, lonquest 801™, Hoe F 3787™,mono-2-ethyl hexyl phosphoric acid (MEHPA), P204™, PC88A™, P507™, or hydroxy-oxime, extractants (e.g. Acorga P50™, Acorga K2000™, LIX 84-I™, SME 529™, LIX 65N™, LIX

64™, LIX 70™, LIX 860™, LIX 622™), or β-diketone metal cation extractants (e.g. LIX 54™, XI-N54™, XI-55™, XI-57™) [Source: Solvent extraction: the coordination chemistry behind extractive metallurgy. Chem. Soc. Rev., 2014, 43, 123].

[00205] For example, the filtered sulfate solution after the co-precipitation of the hydroxide could optionally be purified and/or concentrated before entering the membrane electrolysis.

[00206] For example, the leached solution can be purified before the co-precipitation of the hydroxide. Examples of purification can be related to metals selective separation, e.g. precipitation of hydroxides, precipitation of insoluble salts, oxidative precipitation, ion exchange, solvent extraction, electrochemical plating, crystallization.

[00207] For example, selective precipitation can be performed by addition of e.g. O_2, SO_2 or H_2SO_5, persulfates ($(NH_4)_2S_2O_8$), ammonium oxalate $(NH_4)_2C_2O_4$, chlorine, chlorine compounds (HCl, ClO_2, $HClO_3$), O_3, NaOCl, CoS, Na_2S, NaHS, $CaCO_3$, Na_3PO_4.

[00208] For example, precipitation of hydroxides can be obtained by addition of e.g. LiOH, NaOH, NH_4OH.

[00209] For example, precipitation of insoluble salts can be obtained by addition of dimethylglyoxime.

[00210] For example, the $LiPF_6$ electrolyte can be recovered.

[00211] For example, the solid/liquid (gram of material / volume of liquid) ratio in g/L for the leaching step can be comprise between 1/5 to 1:100.

[00212] For example, the leaching solution can be a mixture of at least one of H_2SO_4, H_2O_2, HNO_3, HCl, nitric acid, citric acid, oxalic acid, aspartic acid, ascorbic acid, glucose.

[00213] For example, the sulfate metals $M(SO_4)$ (with M = Ni, Co, Mn) and/or $Al_2(SO_4)_3$ can be optionally crystallized before being used as precursor in the synthesis of the hydroxide.

[00214] For example, even though the final material was obtained here using co-precipitation method, any other kind of synthesis method leading to the synthesis of a layered oxide material with recycling of lithium-containing sulfate solution is encompassed within the scope of the present disclosure

[00215] According to one example, a process according to the present disclosure is presented in FIG. 1. As it can be seen from FIG. 1, nickel sulfate, cobalt sulfate and manganese sulfate can be mixed together to obtain a composition comprising various metal sulfates. Such a composition can be an aqueous composition, for example, an acidic aqueous composition. For example, a material comprising at least one metal can be leached with H_2SO_4, thereby obtaining the desired metal sulfates composition. Alternatively, various metal sulfates can be reacted with an aqueous acidic composition to obtain the desired metal sulfates composition. LiOH and a chelating agent (for example NH_3) are then added to this mixture to get the mixture formation and eventually to precipitate the desired metal hydroxide. LiOH is a pH enhancer as the sulfate metal reaction starts at high pH, and NH_3 can act as a chelating agent. Once the reaction starts, a solid phase will precipitate (*i.e.* being the hydroxide compound) and can be separated from the liquid phase at high pH, *e.g.* $10 \leq pH \leq 13$. This solid phase precipitate will be further washed with water and dried out at 120°C for 8h under air. Then, the hydroxide phase $NMC(OH)_2$ is obtained. The liquid phase gathered earlier contains dissolved Li_2SO_4, which can be collected after liquid phase filtration. This Li_2SO_4 lithium sulfate can be electrolyzed in a membrane electrolyser into lithium hydroxide LiOH, that could be used as pH enhancer for another mixture formation.

[00216] The person skilled in the art would understand that the electromembrane process can be carried in many different manners and in accordance to various different parameters. For example, such an electromembrane process can be carried as defined in any one of the following references WO2013159194, WO2013177680, WO2014138933, WO 2015/058287, WO 2015/058287, WO 2015/123762, WO2017031595 and WO2018035618.

[00217] The hydroxide phase $NMC(OH)_2$ can be further used to be mixed with LiOH obtained from electrolysis of the Li_2SO_4 to obtain a mixture of metal hydroxides. For example, this mixture of metal hydroxides can be roasted at different temperatures. For example, it can be roasted at a first temperature of 450°C for about 8h under air, then it can be roasted at 800°C for 12h under air. Then, it is crushed and sieved, washed with water, and finally dried at 600°C for about 8h under air. The nickel-manganese-cobalt lithium oxide $Li-(Ni_xMn_yCo_z)O_2$ is then obtained, wherein $0 < x, y, z < 1$ and $x + y + z = 1$. Core-shell materials can also be obtained, with a gradient concentration from the core to the surface for the different metals, as $[LiNi_xM1_yM2_zO_2]_{core}/[LiNi_aM1_bM2_cO_2]_{shell}$, with $x+y+z = 1$, $a+b+c = 1$, M1 = Mn, Co or Al and M2 = Mn, Co or Al, and e.g. $a \neq x$ for Ni being different, leading to the concentration gradient in the final material.

[00218] For example, the metal source can be an at least substantially pure metal leached by the electrochemically generated sulfuric acid.

[00219] For example, the metal source can be a nickel concentrate (containing also cobalt and possibly other elements) leached by the electrochemically generated sulfuric acid.

[00220] For example, the metal source can be a nickel cobalt containing material (e.g. nickel oxide ore, nickel matte, nickel sulfide, mixed sulfide of nickel and cobalt, crude nickel sulfate produced from a copper smelting process, and nickel oxide) leached by the electrochemically generated sulfuric acid.

[00221] For example, the metal source can be an aqueous nickel-cobalt solution such as the solutions referred to as A or B in FIG. 33 or as C or D in FIG. 34 and FIG. 35, leached by the electrochemically generated sulfuric acid.

[00222] For example, the metal source can be an organic solution containing nickel (and cobalt and possibly other elements) that can be stripped by the electrochemically generated sulfuric acid.

[00223] For example, the metal source can be a spent battery leached or constituent thereof (e.g. cathode, anode, black mass, slag, or mixtures thereof) (e.g. the cathode only, or both the anode and the cathode or a black mass, etc) leached by the electrochemically generated sulfuric acid.

[00224] The person skilled in the art would understand that the process shown in FIG. 1 can vary in accordance with the nature of the at least one metal sulfate used as starting material. Various metals can thus be used and various mixtures thereof as starting material.

EXAMPLES

[00225] Synthesis of oxides at high potential for cathode material of lithium ion batteries

[00226] A cathode material was synthetized to produce a lithium transition metal oxide with specific formula, $Li_pNi_xMn_yCo_zAl_qO_2$. The formula has specific percentage to reach certain kind of materials in the industries. The obtained cathodes materials are $LiCoO_2$, $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$, $LiNi_{0.8}Mn_{0.1}Co_{0.1}O_2$ and $LiNi_{0.6}Mn_{0.2}Co_{0.2}O_2$.

Example 1 Synthesis of $Co(OH)_2$

[00227] 28.11g of $CoSO_4{\cdot}7H_2O$ (Strem Chemicals, inc) was dissolved in 100mL of distilled water to produce a solution of 1M (pH around 4-5). 10.49g of $LiOH{\cdot}H_2O$ (Sigma-Aldrich) was dissolved in 250mL of distilled water to obtain a solution of 1M (pH over 12). 5.841mL was taken of a solution of 28-30%vol of ammonia (Sigma-Aldrich) to have a solution of 2M (pH >12).

[00228] The setup was built with a flask round bottom 4-neck (Dima glass inc). One of the neck was used for a nitrogen flow to have an inert atmosphere in the flask. Two other opening were used to pour LiOH and NH_3 and the fourth one was dedicated to the recovery of NH_3 through a condenser.

[00229] The setup was set with the solution of $CoSO_4$ at the bottom of the flask. 10mL of the solution of $CoSO_4$ 1M was first of all deaerated by a flow of nitrogen and the system was maintained under a nitrogen flow for 15 minutes. The temperature was regulated at 60°C. 20mL of NH_3 and 25mL of LiOH were introduced drop by drop and the solution was maintained in the flask with a constant stirring. The reaction began when the pH of the solution reached 10. Once the products reacted (i.e. after 10 minutes), the solution was stirred for another 20 minutes. The substrate was filtered and washed three times with distilled water.

[00230] After filtration, the sample was heated at 120°C for 8 hours. Then, 1g of the $Co(OH)_2$ was collected (pink color). The overall reaction is given Equation 1.

$$CoSO_4 + 2LiOH + NH_3 \rightarrow Co(OH)_2 + Li_2SO_4 + NH_3$$

Equation 1

[00231] In this equation, all the reagents are in aqueous solution. The cobalt hydroxide, product of the reaction in Equation 1, will be used as precursor for the synthesis of the cobalt oxide (cf. example 2). In the mother liquor, an aqueous solution of Li_2SO_4 was mixed with a leftover of LiOH, in excess during the reaction. To convert all the LiOH into lithium sulfate, the solution was neutralized using H_2SO_4 as showed in Equation 2.

$$2LiOH + H_2SO_4 \rightarrow Li_2SO_4 \cdot H_2O + H_2O$$

Equation 2

[00232] The filtrated Li_2SO_4 can be electrolysed and converted into $LiOH \cdot H_2O$. X-ray diffraction was performed on the compound to highlight its high purity.

[00233] FIG. 2 represents the X-Ray diffraction pattern of the $Co(OH)_2$. It may be indexed with the theoretical diffraction peaks of the cobalt hydroxide. Besides, an impurity can be notice, as a small intensity peak is observed at 20°. In the synthesis of the hydroxide, LiOH is used as a source of pH enhancer, as the formation reaction of the hydroxide starts at high pH. For example, LiOH can be replaced by NaOH and X-ray diffraction was performed on the compound to highlight its high purity. In such a case, the electromembrane process can be used for converting Na_2SO_4 into NaOH.

[00234] FIG. 3 represents the X-Ray diffraction pattern of the $Co(OH)_2$ synthesis with NaOH as pH enhancer source. The X-Ray diffraction pattern of the compound may be indexed with the theoretical diffraction peaks of the cobalt hydroxide. Besides, an impurity can be notice, as a small intensity peak is observed at 20° as was observed for the LiOH diffractogram.

[00235] This $Co(OH)_2$ material based on NaOH or LiOH as pH enhancer source was the precursor of various potential products (see below).

Example 2 Synthesis of $LiCoO_2$

[00236] The cobalt hydroxide previously obtained was used as precursors for the synthesis of the lithium cobalt oxide, $LiCoO_2$. Here, the first step was to mix the $LiOH \cdot H_2O$ with the $Co(OH)_2$. This was a stoichiometric reaction as show in Equation 3

$$Co(OH)_2 + LiOH \cdot H_2O + 0.25O_2 \rightarrow LiCoO_2 + 2.5H_2O$$

Equation 3

[00237] The precursors were mixed, crushed, and pellets were done before thermal treatment. These pellets were put in the furnace for 8 hours at 450°C under air. After this step, the pellets were crushed and redone in pellets. The furnace was now set at 800°C for 12 hours under air. The pellets were crushed again and then washed with water. The suspension was filtered, the powder collected and pressed in pellets again. The final step consisted in another thermal treatment for 8 hours at 600°C under air.

[00238] The X-ray diffraction pattern in confirmed the high purity of the lithium cobalt oxide.

[00239] FIG. 4 presents the X-Ray diffraction pattern of the lithium cobalt oxide. One can see an impurity at 20°, that may be a residue of cobalt hydroxide (the same impurity was observed). This impurity has already been reported several times in the literature.

[00240] The lithitated cobalt oxide can also be produced from the cobalt hydroxide obtained with NaOH. The X-Ray diffraction of such compound can be found in FIG. 5 and pointed out that no difference is observed depending on the nature of the base source during the hydroxide synthesis.

[00241] The next step was to characterize the $LiCoO_2$ with the electrochemistry. The cathode electrode was prepared by mixing 83 wt. % of $LiCoO_2$, 9 wt. % of carbon black Timcal^{TM} C65, and 8 wt. % of polyvinylidene difluoride (PVDF) in n-methyl pyrrolidone (NMP) solvent to form a slurry. The slurry was mixed for few hours to homogeneity and spread on a carbon-coated aluminum foil using the doctor blade method. After drying at 70°C in a vacuum oven overnight, electrode disks of 0.5 ± 0.1 mg/cm2 of active material loading were cut and calendered. Standard coin-cells (2032) were assembled in an Ar-filled glove box. Once the electrode was prepared, a lithium foil was used as the anode, 1 M LiPF6 dissolve in ethylene carbonate and diethyl carbonate (1:2 volume ratio) solvents was used as liquid electrolyte. Polypropylene membranes (Celgard inc.) were used as separators. The electrochemical tests were performed on the cells at 30 °C on a VMP electrochemical station (Bio-Logic, France) with cut-off voltages of 3 and 4.3 V vs Li/Li+ at 0.1 C rate for galvanostatic cycling. Three coin cells were prepared per sample to ensure reproducibility of the results. The standard deviation was determined to be ± 1 mAh/g.

[00242] FIG. 6 showed the five first charges and discharges of the $LiCoO_2$. The capacity reached 175 $mAhg^{-1}$ but decrease with the cycling. The capacity of the $LiCoO_2$ change Depending of the potential range but at higher potential, irreversible reaction could happen. However, at 4.3 V, the compound should be stable. Some optimization should be done to optimize the capacity and the stability of the $LiCoO_2$.

Example 3 Synthesis of $Ni_{0.8}Co_{0.15}Al_{0.05}(OH)_2$

[00243] 2.3121g of $NiSO_4{\cdot}6H_2O$ (Strem Chemicals, inc), 0.4628g of $CoSO_4{\cdot}6H_2O$ (Strem Chemicals, inc) and 0.0944g of $Al_2(SO_4)_3{\cdot}H_2O$ (Sigma-Aldrich) were dissolved in 10mL of water.

[00244] The setup and the reaction condition were as described in Example 1. The final product gave $Ni_{0.8}Co_{0.15}Al_{0.05}(OH)_2$ with a green coloration. X-ray diffraction pattern confirmed the formation of the hydroxide, as the diffraction pattern presented in FIG. 7 may fit with the theoretical diffraction pattern of $Ni_{0.8}Co_{0.15}Al_{0.05}(OH)_2$ (vertical bars).

Example 4 Synthesis of $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$

[00245] The next experimental was the formation of the $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$. The experimental procedure was the same as in example 1. X-ray diffraction was used to characterize the formation of the oxide.

[00246] FIG. 8 highlights that the diffraction pattern of the compound may fit with the theoretical diffraction peaks of $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$. The last characterization was the electrochemistry of the compound.

[00247] FIG. 9 showed the charge and discharge of the $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$ at 0.1C rate. The electrochemistry procedure was detailed in example 2. The theoretical capacity of this compound is 279 mAh/g and the specific capacity obtained experimentally was 180 mAh/g. On FIG. 9, one can see two slopes in the discharge curve. This behaviour can be explained by the size particles of the active material, being wide and not optimized for electrochemistry purpose.

Example 5 Synthesis of $Ni_{0.8}Mn_{0.1}Co_{0.1}(OH)_2$

[00248] 2.3131g of $NiSO_4{\cdot}6H_2O$ (Strem Chemicals, inc), 0.3092g of $CoSO_4{\cdot}6H_2O$ (Strem Chemicals, inc) and 0.1859g of $MnSO_4{\cdot}H_2O$ (Sigma-Aldrich) were dissolved in 10 mL of water.

[00249] The setup and the reaction condition were as described in example 1.

[00250] FIG. 10 highlights that the diffraction pattern of the compound may fit with the theoretical diffraction peaks of $Ni_{0.8}Mn_{0.1}Co_{0.1}(OH)_2$.

Example 6 Synthesis of $LiNi_{0.8}Mn_{0.1}Co_{0.1}O_2$

[00251] The next step was the formation of the oxide, $LiNi_{0.8}Mn_{0.1}Co_{0.1}O_2$. The experimental set-up was the same as in example 2. X-ray diffraction was used to characterize the formation of the oxide.

[00252] FIG. 11 highlights that the diffraction pattern of the compound may fit with the theoretical diffraction peaks of $LiNi_{0.8}Mn_{0.1}Co_{0.1}O_2$.

[00253] The capacity reached 175 $mAhg^{-1}$ but decrease with the cycling. The capacity of the $LiCoO_2$ change depending of the potential range but at higher potential, irreversible reaction could happen.

Example 7 Synthesis of $Ni_{0.6}Mn_{0.2}Co_{0.2}(OH)_2$

[00254] 1.7348g of $NiSO_4{\cdot}6H_2O$ (Strem Chemicals, inc), 0.6184g of $CoSO_4{\cdot}6H_2O$ (Strem Chemicals, inc) and 0.3674g of $MnSO_4{\cdot}H_2O$ (Sigma-Aldrich) was dissolved in 10 mL of water.

[00255] The setup and the reaction condition were as described in example 1.

[00256] FIG. 12 shows highlights that the diffraction pattern of the compound may fit with the theoretical diffraction peaks of $Ni_{0.6}Mn_{0.2}Co_{0.2}(OH)_2$.

Example 8 Synthesis of $LiNi_{0.6}Mn_{0.2}Co_{0.2}O_2$

[00257] The next step was the formation of the oxide, $LiNi_{0.6}Mn_{0.2}Co_{0.2}O_2$. The experimental step was the same as the example 2. X-ray diffraction was used to characterize the formation of the oxide.

[00258] FIG. 13 highlights that the diffraction pattern of the compound may fit with the theoretical diffraction peaks of $LiNi_{0.6}Mn_{0.2}Co_{0.2}O_2$.

[00259] FIG. 14 represents the charge/discharge curves of LiNi0.6Mn0.2Co0.2O2 at 0.1C rate. The electrochemistry instrument and method were as described in example 2. The theoretical capacity of this compound is 275 mAh/g and the specific capacity obtained experimentally was 170 mAh/g. On FIG. 14, one can see two slopes in the discharge curve. This behaviour can be explained by the size particles of the active material, being wide and not optimized for electrochemistry purpose.

Example 9 Electrolysis of lithium sulfate and conversion into lithium hydroxide

[00260] Electrolysis of lithium sulfate was carried out in a two-compartment cell ICI FM-21 (similar to the cell of FIG. 2 of WO2015058287) by following the general procedure described in Example 1 of WO2015058287. The experimental conditions were as follows :

Cell : FM-21 2400 cm^2

Current density : 4.0 kA/m^2

Temperature : 60 °C

Li_2SO_4 : 300 g/L (batch)

$LiOH \cdot H_2O$:2 M

[00261] The results obtained were as follows:

Conversion rate : 40 %

H_2SO_4 : 10.2 %

Current efficiency : 76.9 %

Flow rate LiOH : 14.4 L/h

Productivity : 4.75 kg of $LiOH \cdot H_2O/h/m^2$

Voltage (at the cell): 4.39 V

Energy : 3678 kWh/TM $LiOH \cdot H_2O$

[00262] FIGS. 15 to 22 show the results obtained during electrolysis of Li_2SO_4.

[00263] FIG. 15 is a plot showing sulphuric acid concentration in the anolyte stream as a function of batch time; FIG. 16 is a plot showing anolyte and catholyte conductivities as a function of batch time; FIG. 17 is a plot showing anolyte and catholyte temperature as a function of batch time; FIG. 18 is a plot showing voltage at cell and at current generator as a function of batch time; FIG. 19 is a plot showing productivity in milliliters of lithium hydroxide monohydrate equivalent per minute as a function of sulfuric acid concentration in the anolyte; FIG. 20 is a plot showing productivity in liters of lithium hydroxide monohydrate equivalent per hour as a function of sulfuric acid concentration in the anolyte; FIG. 21 is a plot showing current efficiency as a function of sulfuric acid concentration in the anolyte; FIG. 22 is a plot showing productivity in kilograms of lithium hydroxide monohydrate equivalent per hour per meter square of electroactive area as a function of sulfuric acid concentration in the anolyte; FIG. 23 is a plot showing electric energy consumption related to the electrochemical conversion in kilowatt-hour per metric ton of lithium hydroxide monohydrate equivalent as a function of sulfuric acid concentration in the anolyte.

[00264] As shown in FIG. 24 LiOH can be added as a source of pH enhancer to a mixture of metal sulfate(s) for the precipitation of the metal hydroxide(s). After precipitation of the metal hydroxide(s), Li_2SO_4 can be recovered as a dissolved species in aqueous solution to be inserted in the membrane electrolyser, and can be converted into LiOH and optionally going through evaporation, crystallization and drying before reacting with a metal hydroxide(s) to form a metal oxide(s). Sulfuric acid is used for the leaching of the transition metal source, generating metals as dissolved species in sulfate forms.

[00265] As shown in FIG. 25 NaOH can be added as a source of pH enhancer to a mixture of metal sulfate(s) for the precipitation of the metal hydroxide(s). After precipitation of the metal hydroxide(s), Na_2SO_4 can be recovered as a dissolved species in aqueous solution to be inserted in the membrane electrolyser. LiOH can be reacted with a metal hydroxide(s) to form a metal oxide(s). If Lithium is present in the Transition Metal Source, it will be carried out in the metal sulfate solution, the obtained Li2SO4 can be separated from Na2SO4 to purify the Na2SO4 solution before being inserted in the membrane electrolyser. The LiOH used to react with the Lithiated Metal oxide can come from another electromembrane process, or be a commercial LiOH.

[00266] As shown in FIG. 26, a mixture of NaOH and LiOH is used as a source of pH enhancer to a mixture of metal sulfate(s) for the precipitation of the metal hydroxide(s). After precipitation of the metal hydroxide(s), a mixture of Li_2SO_4 and Na_2SO_4 can be recovered as dissolved species in aqueous solution to be inserted in the membrane electrolyser, and Li_2SO_4 can be converted into LiOH to react with a metal hydroxide(s) to form a metal oxide(s). LiOH can be separated from NaOH. For example, LiOH can be substantially selectively precipitated (for example via evaporation, crystallization and drying step) over NaOH and thus separated therefrom. Also, Li2SO4 can be optionally separated from Na2SO4 before reacting in the electromembrane process. The obtained LiOH can be reacted with so as to eventually be used to generate metal oxide(s) by reacting with the metal hydroxide(s) to form a metal oxide(s).

[00267] As shown in FIG. 27 NaOH can be added as a source of pH enhancer to a mixture of metal sulfate(s) for the precipitation of the metal hydroxide(s). For example, NaOH can be used instead of LiOH as a pH enhancer because of economical reasons. After precipitation of the metal hydroxide(s), a mixture of Li_2SO_4 and Na_2SO_4 can be recovered as a dissolved species in aqueous solution to be inserted in the membrane electrolyser, and Li_2SO_4 can be converted into LiOH to react with a metal hydroxide(s) to form a metal oxide(s). LiOH can be substantially selectively precipitated (for example via evaporation, crystallization and drying step) over NaOH. The obtained LiOH can be reacted with so as to eventually be used to generate metal oxide(s) by reacting with the metal hydroxide(s) to form a metal oxide(s).The person skilled in the art would understand that if for example the transition metal source is a spent battery, it can be possible that 100 % of the lithium contained therein will not necessarily be electrolyzed and thus, an external source of Li_2SO_4 can be provided to the electromembrane process to be converted into LiOH.

[00268] As shown in FIG. 28, the LiOH used for the precipitation of the hydroxide can be optionally crystallized. Moreover, the lithium sulfate solution can be purified and concentrated before being inserted in electromembrane process. For example, an external source of Li_2SO_4 can be provided in the present case. In fact, since LiOH generated is used for (i) reacting with the metal sulfate and (ii) to be mixed with the obtained metal hydroxide(s), an external source of Li_2SO_4 can be provided.

[00269] As shown in FIG. 29, the electrochemically generated sulfuric acid solution, called the anolyte solution, can be concentrated to leach the transition metal source, e.g. a battery active material.

[00270] The anolyte concentration process described in FIG. 29 can be carried out by a method or process as described in any one of WO2015123762, WO2017031595 and WO2018035618. The person skilled of the art can understand that the anolyte concentration from FIG. 29 can therefore be applied in any of FIG. 24 to FIG. 28. The Anolyte solution after concentration will be lithium-depleted, and the Li2SO4 rich solution will be inserted back in the electromembrane system to be processed. The lithium sulfate solution obtained after anolyte concentration can be mixed with the Li_2SO_4 solution recovered after hydroxide precipitation as described in FIG. 29. Such mixture of Li_2SO_4 solutions can be returned to the electromembrane process.

[00271] FIG. 30 describes the synthesis of a core-shell design material. The metal hydroxide with a core-shell design can be precipitated as described from FIG. 24 to FIG. 29, and the lithiated material core-shell oxide can be obtained after addition of LiOH.

[00272] From FIG. 28 to FIG. 29, the person skilled of the art can understand that LiOH optionally crystallized can be replaced by NaOH or a mixture of both to enhance the pH. Same apply for Li2SO4 that could be replaced by Na_2SO_4 or a mixture of both. The person skilled of the art can also understand that the concentration and purification of the sulfate solution as describe in FIG. 29 can be applied for any processes from FIG. 24 to FIG. 30.

[00273] FIG. 31 describes the precipitation of metal carbonates instead of metal hydroxides. To do so, the LiOH as generated from the membrane electrolysis can be carbonated to form Li_2CO_3. For example, carbonatation can be carried out as described in WO2013177680, WO2006104367, WO2018134536 or in WO2015058287. The lithium carbonate can react with the metal sulfate to form the metal carbonate. A lithium sulfate solution will be recovered as described in Fig. 24 to 30.

[00274] The person skilled of the art will understand that all the possible embodiment described from FIG. 24 to FIG. 31 can also be applied in FIG. 32 replacing sulfate by nitrate (e.g. concentration of Li_2SO_4 solution and/or Li_2SO^4 and mixture with Na_2SO_4).

[00275] Besides, the person skilled of the art can understand that LiOH can be replaced by NaOH or a mixture of both in any of FIG. 24 to FIG. to FIG. 31. Same apply for Li2SO4 that could be replaced by Na2SO4 or a mixture of both.

[00276] From FIG. 24 to FIG. 31, various sources of acid solution can be used for the reaction with the Transition Metal source, for example it can be sulfuric acid solution (Fig. 24), lithium sulfate solution (Fig. 29), and anolyte solution. For example, these varius sources of acid solution for the leaching solution can be: (A) Electrochemically generated sulfuric acid solution, called anolyte solution; (B) Partially concentrated sulfuric acid solution generated by membrane electrolysis, called (diluted) Lithium Sulfate solution or (C) Sulfuric acid. The (a) anolyte solution relates to an electrochemically generated sulfuric acid solution from a membrane electrolysis, having the chemical composition as presented in Table 1. The concentration of this solution was ≈ 1.5 M H_2SO_4.

	Percentages (wt%)
Li_2SO_4	10-20
H_2SO_4	10-15
H_2O	65-75

Table 1: Composition of the electrochemically generated sulfuric acid solution as it exists in the membrane electrolysis.

[00277] The (b) partially concentrated sulfuric acid solution generated by membrane electrolysis, called (diluted) Lithium Sulfate solution, consists of the previous anolyte solution depleted in lithium, concentrated and then diluted in water to reach a concentration of ≈ 1 M H_2SO_4.

[00278] From FIG. 32, nitric acid can be generated from the salt splitting of LiNO3 used instead of Li2SO4. The leaching of the transition metal source with nitric acid will lead to the production of metal nitrates dissolved in solutions. Then, LiOH is added for the precipitation of the hydroxide, and a nitric lithiated solution can be filtrate. This LiNO3 solution can enter the electromembrane process to be converted into LiOH and HNO3. All the embodiments of FIG. 24 to FIG. 31 apply here when replacing sulfate by FIG. 32.

[00279] The overall protocol starting from the Ni and Co concentrate is illustrated in FIG. 33, FIG. 34, FIG. 35, and can lead to the production of high purity Co and Ni aqueous phases (called solution A and B in FIG. 33, FIG. 34, FIG. 35), to the production of high purity Co or Ni aqueous solutions (called C and D), or to the cobalt sulfate or nickel sulfate crystallized salts (called E and F). . For example, the pH is increased from solution A to B to ensure a maximum recovery of the cobalt in the organic phase. For example, Lithium Sulfate solution can be provided by the anolyte solution as generated by the electromembrane process, and concentrated as described in FIG. 29

[00280] From FIG. 33 to FIG. 35, various sources of acid solution can be used for the leaching of the Li-Co concentrate, for example it can be sulfuric acid solution (FIG. 33), lithium sulfate solution (FIG. 34), and anolyte solution (FIG. 35).

[00281] The person skilled in the art would understand that for example the embodiments provided in FIG. 33 to FIG. 35 can be applicable in the processes shown FIG. 24 to FIG. 31, and metal sulfates obtained in FIG. 33 to FIG. 35 can be the source of the Transition Metal box from Fig. 24 to Fig. 31. Besides, the sulfate acid source used for the leaching in FIG. 33 to FIG. 35 could be replaced by nitric to obtain a transition metal source in form of a nitrate as described in FIG. 32.

[00282] The person skilled of the art would understand that LiOH used in FIG. 24 to FIG. 30 and in FIG. 34 to react with the metal hydroxide / carbonate to from the lithiated metal oxide(s) can be carbonated, generating Li_2CO_3 reacting with the metal hydroxide / carbonate to form the lithiated metal oxide(s). Other carbonates as described in the present disclosure can also be used such as Na_2CO_3, K_2CO_3, Rb_2CO_3, Cs_2CO_3, $MgCO_3$, $CaCO_3$, $SrCO_3$ or $BaCO_3$.

[00283] The person skilled of the art would understand that nitrates used in FIG. 35 can be an alternative to sulfates as presented in FIG. 24 to FIG. 34, and all the processes presented in FIG. 24 to FIG. 34 can be used replacing sulfates by nitrates.

[00284] For example, conversion from metal carbonates to lithium oxide is described in WO2006104367.

[00285] For example, the electrochemically generated sulfuric acid (H_2SO_4 solution) generated in FIGS. 24 to 28 can contain lithium sulfate, sodium sulfate and/or potassium sulfate. H_2SO_4 can be separated from lithium sulfate, sodium sulfate and/or potassium sulfate as shown in FIG. 29 through anolyte concentration. For example, such a separation can be achived by a selective crystallization of a sulfate monohydrate. For example, anolyte concentration can be carried out by selective sulfate precipitation as defined in any one of WO2015123762, WO2017031595 and WO2018035618.

[00286] Besides, the person skilled of the art can understand that the acid solution generated by electromembrane process in FIG. 24 to FIG. 35 can be replaced by the anolyte solution and a concentration step as presented in FIG. 29.

[00287] The person skilled of the art will understand that all the possible embodiment described from FIG. 24 to FIG. 34 can also be applied in FIG. 35 (e.g. concentration of Li_2SO_4 solution and/or Li_2SO_4 and mixture with Na_2SO_4).

Example 10 – Core-shell synthesis

[00288] For the synthesis of a gradient concentration material with a composition $Li[Ni_dM1_eM2_f]O_2$ with d+e+f = 1, being made of a core $[LiNi_xM1_yM2_zO_2]$ with x+y+z = 1 and a shell $[LiNi_aM1_bM2_cO_2]$ with a+b+c = 1, with M1 = Mn, Co or Al and M2 = Mn, Co or Al and with $x<d<a$, $y<e<b$, $z<f<c$.

In order to prepare such a spherical Core-Shell material, the hydroxide precursor has to be obtained first, and can be synthetized via co-precipitation. In such a synthesis method, a certain amount of $NiSO_4{\cdot}6H2O$ (and optionally M1 at a given concentration and M2 at a different concentration) aqueous solution was used as a starting material for the core composition of $Ni_xM1_yM2_z(OH)_2$. The metal aqueous solution were continuously fed into a batch reactor already filled with certain amounts of deionized water, $NaOH_{(aq.)}$ as pH enhancer and $NH_4OH_{(aq.)}$ as chelating agent, under a nitrogen atmosphere. Simultaneously, NaOH at a given concentration and adequate amount of $NH4OH_{(aq.)}$ were pumped into the reactor. Once the precursor $Ni_xM1_yM2_z(OH)_2$ is formed in solution, the second solution, an aqueous solution of the desired metals $Ni_aM1_bM2_c(OH)_2$ (e.g. M1 and M2 = Ni, Mn, Co, Al) was introduced into the reactor. The obtained $Ni_dM1_eM2_f(OH)_2$ (with $x<d<a$, $y<e<b$, $z<f<c$) powders were filtered, washed, and dried under vacuum at 110oC for 12 h. To prepare $Li[Ni_dM1_eM2_f]O_2$, the precursor $Ni_dM1_eM2_f(OH)_2$ was mixed with $LiOH{\cdot}H2O$ and calcined at 700°C for 10 h under oxygen atmosphere.

[00289] For example, the metal source can be a spent battery leached or constituent thereof (e.g. cathode, anode, black mass, slag or mixtures thereof) (e.g. the cathode only, or both the anode and the cathode or a black mass, etc) leached by the electrochemically generated sulfuric acid.

[00290] The leaching metal sulfate solution can contain the metal retrieved from the spent battery (e.g. Li, Ni, Co and/or Al and/or Mn). For example, NaOH can be added as a source of pH enhancer to a mixture of metal sulfate(s) for the precipitation of the metal hydroxide(s). After precipitation of the metal hydroxide(s), a mixture of Li_2SO_4 and Na_2SO_4 can be recovered as a dissolved species in aqueous solution to be inserted in the membrane electrolyser, and Li_2SO_4 can be converted into LiOH to react with a metal hydroxide(s) to form a metal oxide(s).

[00291] The person skilled in the art would understand that another base could be used instead of NaOH. For example, KOH, RbOH, CsOH, $Mg(OH)_2$, $Ca(OH)_2$, $Sr(OH)_2$, or $Ba(OH)_2$ could be used.

[00292] The embodiments of paragraphs [0036] to [00291] of the present disclosure are presented in such a manner in the present disclosure so as to demonstrate that every combination of embodiments, when applicable can be made. These embodiments have thus been presented in the description in a manner

thereby demonstrating that they can be combined together in all possible manners. For example, all the possible combination, when applicable, between the embodiments of paragraphs [0036] to [00291] and the processes of paragraphs [0005] to [0035] are hereby covered by the present disclosure.

[00293] The present disclosure has been described with regard to specific examples. The description was intended to help the understanding of the disclosure, rather than to limit its scope. It will be apparent to one skilled in the art that various modifications can be made to the disclosure without departing from the scope of the disclosure as described herein, and such modifications are intended to be covered by the present document.

[00294] Where a term in the present disclosure is found to be defined differently in a document cited herein, the definition provided herein is to serve as the definition for the term.

WHAT IS CLAIMED IS:

1. A process for preparing a metal hydroxide for use in the manufacture of a cathode material for lithium ion batteries comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum, said process comprising:

reacting a metal sulfate comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum with lithium hydroxide and optionally a chelating agent in order to obtain a solid comprising said metal hydroxide and a liquid comprising lithium sulfate;

separating said liquid and said solid from one another to obtain said metal hydroxide;

submitting said liquid comprising lithium sulfate to an electromembrane process for converting said lithium sulfate into lithium hydroxide; and

reusing said lithium hydroxide obtained by said electromembrane process for reacting with said metal sulfate.

2. A process for preparing a metal hydroxide comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum, said process comprising:

reacting a metal sulfate comprising (i) lithium; (ii) at least one metal chosen from nickel and cobalt and optionally (iii) at least one metal chosen from manganese and aluminum with sodium hydroxide and optionally a chelating agent in order to obtain a solid comprising said metal hydroxide and a liquid comprising sodium sulfate and lithium sulfate;

separating said liquid and said solid from one another to obtain said metal hydroxide;

submitting said liquid comprising sodium sulfate and lithium sulfate to an electromembrane process for converting said sodium sulfate and said lithium sulfate into sodium hydroxide and lithium hydroxide; and

reusing said sodium hydroxide obtained by said electromembrane process for reacting with said metal sulfate.

3. The process of claim 2, wherein LiOH is substantially selectively crystallized by evaporative crystallization.

4. The process of claim 2, wherein LiOH is substantially selectively crystallized and removed from said electromembrane process by evaporative crystallization.

5. The process of claim 2, wherein LiOH is separated from NaOH by substantially selectively crystallizing LiOH over NaOH.

6. The process of claim 2, wherein LiOH is separated from NaOH by substantially selectively crystallizing LiOH by evaporative crystallization.

7. The process of any one of claims 1 to 6, wherein said solid is a precipitate comprising said metal hydroxide, said precipitate being obtained at a pH of about 8 to about 14.

8. The process of any one of claims 1 to 6, wherein said solid is a precipitate comprising said metal hydroxide, said precipitate being obtained at a pH of about 9 to about 13 or about 10 to about 12.

9. The process of any one of claims 1 to 8, further comprising washing said metal hydroxide.

10. The process of claim 9, further comprising drying said metal hydroxide.

11. The process of claim 10, wherein drying comprises drying at a temperature of at least 80 °C for a period of time of about 1 to about 4 hours.

12. The process of any one of claims 1 to 11, wherein said chelating agent is present.

13. The process of claim 12, wherein said chelating agent is ammonia.

14. A process for preparing a metal oxide comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum, said process comprising:

reacting a metal sulfate comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum with lithium hydroxide and optionally a chelating agent to obtain a solid comprising a metal hydroxide comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum, and a liquid comprising lithium sulfate;

separating said liquid and said solid from one another to obtain said metal hydroxide;

submitting said liquid comprising lithium sulfate to an electromembrane process for converting said lithium sulfate into lithium hydroxide; and

reusing at least a first portion of said lithium hydroxide obtained by said electromembrane process for reacting with said metal sulfate;

reacting at least a second portion of said lithium hydroxide obtained by said electromembrane process with said obtained metal hydroxide to obtain a mixture of metal hydroxides; and

roasting said mixture of metal hydroxides to obtain said metal oxide.

15. A process for preparing a metal oxide comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum, said process comprising:

reacting a metal sulfate comprising (i) lithium; (ii) at least one metal chosen from nickel and cobalt and optionally (iii) at least one metal chosen from manganese and aluminum with sodium hydroxide and optionally a chelating agent in order to obtain a solid comprising a metal hydroxide and a liquid comprising sodium sulfate and lithium sulfate;

separating said liquid and said solid from one another to obtain said metal hydroxide;

submitting said liquid comprising sodium sulfate and lithium sulfate to an electromembrane process for converting said sodium sulfate and said lithium sulfate into sodium hydroxide and lithium hydroxide;

separating said lithium hydroxide and said sodium hydroxide from one another;

reusing at least a first portion of said sodium hydroxide obtained by said electromembrane process for reacting with said metal sulfate;

reacting at least a first portion of said lithium hydroxide obtained by said electromembrane process with said obtained metal hydroxide to obtain a mixture of metal hydroxides; and

roasting said mixture of metal hydroxides to obtain said metal oxide.

16. The process of claim 14 or 15, wherein said solid is a precipitate comprising said metal hydroxide, said precipitate being obtained at a pH of about 9 to about 14.

17. The process of claim 14 or 15, wherein said solid is a precipitate comprising said metal hydroxide, said precipitate being obtained at a pH of about 10 to about 13.

18. The process of claim 14 or 15, wherein said solid is a precipitate comprising said metal hydroxide, said precipitate being obtained at a pH of about 10.5 to about 12.5.

19. The process of any one of claims 14 to 18, further comprising washing said metal hydroxide.

20. The process of claim 19, further comprising drying said metal hydroxide.

21. The process of any one of claims 14 to 20, wherein said metal sulfate is reacted with said hydroxide and a chelating agent that is chosen from ammonia, NH_4OH, acetylacetone, 5-sulfosalicylic acid, oxalic acid, EDTA (ethylenediaminetetraacetic acid), NTA (nitrilotriacetic acid), DCTA (trans-1,2-diaminocyclohexanetetraacetic acid), DTPA (diethylenetriaminepentaacetic acid), and EGTA (ethylene glycol bis(2-aminoethyl ether)-N,N,N′,N′-tetraacetic acid).

22. The process of any one of claims 14 to 21, wherein roasting said mixture of metal hydroxides comprises roasting at a first temperature of at least 350 °C for a period of time of at least 4 hours.

23. The process of any one of claims 14 to 21, wherein roasting said mixture of metal hydroxides comprises roasting at a first temperature of at least 400 °C for a period of time of at least 6 hours.

24. The process of claim 22 or 23, wherein roasting said mixture of metal hydroxides further comprises roasting at a second temperature of at least 600 °C for a period of time of at least 6 hours.

25. The process of claim 22 or 23, wherein roasting said mixture of metal hydroxides further comprises roasting at a second temperature of at least 700 °C for a period of time of at least 8 hours.

26. The process of any one of claims 1 to 25, wherein said electromembrane process comprises an electrolysis membrane process.

27. The process of any one of claims 1 to 25, wherein said electromembrane process comprises a two-compartment monopolar or bipolar membrane electrolysis process.

28. The process of claim 27, wherein said two-compartment monopolar or bipolar membrane electrolysis process is carried out in a first electrochemical cell comprising an anolyte compartment separated from a catholyte compartment by a cation exchange membrane.

29. The process of claim 28, wherein said cation exchange membrane comprises a perfluorinated sulfonic acid.

30. The process of claim 28 or 29 wherein during said two-compartment monopolar or bipolar membrane electrolysis process, an aqueous stream comprising lithium sulfate is introduced into said anolyte compartment, a first lithium-reduced aqueous stream is removed from said anolyte compartment and a first lithium hydroxide-enriched aqueous stream is removed from said catholyte compartment.

31. The process of claim 30, wherein during said two-compartment monopolar or bipolar membrane electrolysis process, conversion of said lithium sulfate to lithium hydroxide proceeds until hydroxide current efficiency is no longer at least substantially maintained so that it decreases.

32. The process of claim 30, wherein during said two-compartment monopolar or bipolar membrane electrolysis process, conversion of said lithium sulfate to lithium hydroxide proceeds until pH in said anolyte compartment is a value of from about 0.4 to about 1.0.

33. The process of claim 30, wherein during said two-compartment monopolar or bipolar membrane electrolysis process, conversion of said lithium sulfate to lithium hydroxide proceeds until pH in said anolyte compartment is a value of about 0.5 to about 0.7.

34. The process of any one of claims 30 to 33, wherein in said catholyte compartment of said two-compartment monopolar or bipolar membrane electrolysis process, lithium hydroxide is at least substantially maintained at a concentration of about 1 M to about 4 M.

35. The process of any one of claims 30 to 33, wherein in said catholyte compartment of said two-compartment monopolar or bipolar membrane electrolysis process, lithium hydroxide is at least substantially maintained at a concentration of about 2 M to about 3 M.

36. The process of any one of claims 30 to 35, wherein during said two-compartment monopolar or bipolar membrane electrolysis process, said aqueous stream comprising lithium sulfate is introduced into said anolyte compartment at a temperature of about 20 °C to about 100 °C.

37. The process of any one of claims 30 to 35, wherein during said two-compartment monopolar or bipolar membrane electrolysis process, said aqueous stream comprising lithium sulfate is introduced into said anolyte compartment at a temperature of about 40 °C to about 70 °C.

38. The process of any one of claims 30 to 35, wherein during said two-compartment monopolar or bipolar membrane electrolysis process, said aqueous stream comprising lithium sulfate is introduced into said anolyte compartment at a temperature of about 45 °C to about 55 °C.

39. The process of any one of claims 30 to 35, wherein during said two-compartment monopolar or bipolar membrane electrolysis process, said aqueous stream comprising lithium sulfate and is introduced into said anolyte compartment at a temperature of about 50°C.

40. The process of any one of claims 30 to 35, wherein during said two-compartment monopolar or bipolar membrane electrolysis process, said first lithium-reduced aqueous stream is removed from said anolyte compartment at a temperature of about 50 °C to about 85 °C.

41. The process of any one of claims 30 to 35, wherein during said two-compartment monopolar or bipolar membrane electrolysis process, said aqueous stream comprising lithium sulfate is introduced into said anolyte compartment at a temperature of about 55 °C to about 65 °C.

42. The process of any one of claims 30 to 41, wherein during said two-compartment monopolar or bipolar membrane electrolysis process, said first lithium-reduced aqueous stream is removed from said anolyte compartment at a temperature of about 60°C.

43. The process of any one of claims 28 to 42, wherein during said two-compartment monopolar or bipolar membrane electrolysis process, temperature in said first electrochemical cell is at least substantially maintained at a value of about 50 °C to about 85 °C.

44. The process of any one of claims 28 to 43, wherein in said two-compartment monopolar or bipolar membrane electrolysis process, current density is at least substantially maintained at a value of from about 0.5 kA/m^2 to about 6 kA/m^2.

45. The process of any one of claims 28 to 43, wherein in said two-compartment monopolar or bipolar membrane electrolysis process, current density is at least substantially maintained at a value of from about 3 kA/m^2 to about 5 kA/m^2.

46. The process of any one of claims 28 to 43, wherein in said two-compartment monopolar or bipolar membrane electrolysis process, voltage is at least substantially maintained at a value of about 3 V to about 8 V.

47. The process of any one of claims 28 to 43, wherein in said two-compartment monopolar or bipolar membrane electrolysis process, voltage is at least substantially maintained at a value of from about 5 V to about 10 V.

48. The process of any one of claims 28 to 47, wherein said first electrochemical cell has a cell area of about 0.2 m^2 to about 4 m^2.

49. The process of any one of claims 28 to 47, wherein said first electrochemical cell has a cell area of about 0.5 m^2 to about 3.5 m^2.

50. The process of any one of claims 28 to 47, wherein said first electrochemical cell has a cell area of about 1 m^2 to about 3 m^2.

51. The process of any one of claims 28 to 47, wherein said first electrochemical cell has a cell area of about 1 m^2 to about 2 m^2.

52. The process of any one of claims 1 to 25, wherein said electromembrane process comprises an electrodialysis process.

53. The process of any one of claims 1 to 25, wherein said electromembrane process comprises a three-compartment monopolar or bipolar membrane electrolysis process.

54. The process of claim 53, wherein said three-compartment monopolar or bipolar membrane electrolysis process is carried out in a second electrochemical cell comprising an anolyte compartment separated from a central compartment by an anion exchange membrane and a catholyte compartment separated from said central compartment by a cation exchange membrane.

55. The process of claim 54, wherein said cation exchange membrane comprises a sulfonated polytetrafluoroethylene.

56. The process of claim 54 or 55, wherein during said three-compartment monopolar or bipolar membrane electrolysis process, a first lithium-reduced aqueous stream is introduced into said central compartment, a second lithium-reduced aqueous stream is removed from said central compartment and a second lithium hydroxide-enriched aqueous stream is removed from said catholyte compartment.

57. The process of claim 56, wherein said three-compartment monopolar or bipolar membrane electrolysis process further comprises producing sulfuric acid in said anolyte compartment and removing a sulfuric acid-containing aqueous stream from said anolyte compartment.

58. The process of claim 57, wherein in said three-compartment monopolar or bipolar membrane electrolysis process, said anion exchange membrane is a proton-blocking membrane.

59. The process of claim 57 or 58, wherein in said anolyte compartment of said three-compartment monopolar or bipolar membrane electrolysis

process, said sulfuric acid is at least substantially maintained at a concentration of sulfuric acid of about 0.1 M to about 2 M.

60. The process of claim 57 or 58, wherein in said anolyte compartment of said three-compartment monopolar or bipolar membrane electrolysis process, said sulfuric acid is at least substantially maintained at a concentration of sulfuric acid of less than or equal to about 0.7 M to about 1.2 M.

61. The process of any one of claims 56 to 60, wherein in said catholyte compartment of said three-compartment membrane electrolysis process, said lithium hydroxide is at least substantially maintained at a concentration of about 1 M to about 4 M.

62. The process of any one of claims 56 to 60, wherein in said catholyte compartment of said three-compartment membrane electrolysis process, said lithium hydroxide is at least substantially maintained at a concentration of about 2 M to about 3 M.

63. The process of any one of claims 56 to 62, wherein during said three-compartment monopolar or bipolar membrane electrolysis process, said first lithium-reduced aqueous stream is introduced into said central compartment at a temperature of about 40 °C to about 85 °C.

64. The process of any one of claims 56 to 62, wherein during said three-compartment monopolar or bipolar membrane electrolysis process, said second lithium-reduced aqueous stream is removed from said central compartment at a temperature of about 40 °C to about 80 °C.

65. The process of any one of claims 56 to 62, wherein during said three-compartment monopolar or bipolar membrane electrolysis process, temperature in said second electrochemical cell is at least substantially maintained at a value of about 50°C to about 70°C.

66. The process of any one of claims 56 to 65, wherein in said three-compartment monopolar or bipolar membrane electrolysis process,

current density is at least substantially maintained at a value of about 0.5 kA/m^2 to about 5 kA/m^2.

67. The process of any one of claims 56 to 65, wherein in said three-compartment monopolar or bipolar membrane electrolysis process, current density is at least substantially maintained at a value of about 1 kA/m^2 to about of about 10 kA/m^2.

68. The process of any one of claims 56 to 65, wherein in said three-compartment monopolar or bipolar membrane electrolysis process, voltage is at least substantially maintained at a value of about 5 V to about 9 V.

69. The process of any one of claims 56 to 68, wherein said electrochemical cell has a cell area of about 0.2 m^2 to about 4 m^2.

70. The process of any one of claims 56 to 68, wherein said electrochemical cell has a cell area of about 0.5 m^2 to about 3.5 m^2.

71. The process of any one of claims 56 to 68, wherein said electrochemical cell has a cell area of about 1 m^2 to about 3 m^2.

72. The process of any one of claims 56 to 68, wherein said electrochemical cell has a cell area of about 1 m^2 to about 2 m^2.

73. The process of claim 56, wherein said three compartment monopolar or bipolar membrane electrolysis process further comprises introducing ammonia into said anolyte compartment, producing ammonium sulfate in said anolyte compartment and removing an ammonium sulfate-containing aqueous stream from said anolyte compartment.

74. The process of claim 73, wherein in said three-compartment monopolar or bipolar membrane electrolysis process, said anion exchange membrane is not a proton-blocking membrane.

75. The process of claim 74, wherein said anion exchange membrane is an Astom^{TM} AHA membrane.

76. The process of any one of claims 73 to 75, wherein in said anolyte compartment of said three-compartment monopolar or bipolar membrane electrolysis process, said ammonium sulfate is at least substantially maintained at a concentration of ammonium sulfate of about 0.5 M to about 4 M.

77. The process of any one of claims 73 to 75, wherein in said anolyte compartment of said three-compartment monopolar or bipolar membrane electrolysis process, said ammonium sulfate is at least substantially maintained at a concentration of ammonium sulfate of about 3 M.

78. The process of any one of claims 73 to 77, wherein in said catholyte compartment of said three-compartment monopolar or bipolar membrane electrolysis process, said lithium hydroxide is at least substantially maintained at a concentration of about 1 M to about 3 M.

79. The process of any one of claims 73 to 77, wherein in said catholyte compartment of said three-compartment monopolar or bipolar membrane electrolysis process, said lithium hydroxide is at least substantially maintained at a concentration of about 2 M.

80. The process of any one of claims 1 to 25, wherein said electromembrane process is electrolysis.

81. The process of any one of claims 1 to 25, wherein said electromembrane process is electrodialysis.

82. The process of any one of claims 1 to 81, wherein the chelating agent is chosen from NH_3, NH_4OH, acetylacetone, 5-sulfosalicylic acid, and oxalic acid.

83. The process of any one of claims 1 to 81, wherein the chelating agent is chosen from EDTA (ethylenediaminetetraacetic acid), NTA (nitrilotriacetic acid), DCTA (trans-1,2-diaminocyclohexanetetraacetic acid), DTPA (diethylenetriaminepentaacetic acid), and EGTA (ethylene glycol bis(2-aminoethyl ether)-N,N,N′,N′-tetraacetic acid).

84. The process of claim 83, wherein said chelating agent is present.

85. The process of any one of claims 1 to 84, wherein said metal hydroxide is $NiCoAl(OH)_2$ or $NiMnCo(OH)_2$.

86. The process of any one of claims 1 to 84, wherein said metal hydroxide is chosen from $Ni_{0.8}Co_{0.15}Al_{0.05}(OH)_2$, $Ni_{0.8}Mn_{0.1}Co_{0.1}(OH)_2$ and $Ni_{0.6}Mn_{0.2}Co_{0.2}(OH)_2$.

87. The process of any one of claims 14 to 25, wherein said metal oxide is of formula $LiMO_2$, or $Li_{(1+x)}M_{(1-x)}O_2$ for lithium-rich and $Li_{(1-z)}M_{(1+z)}O_2$ for Li-deficient, wherein M is at least one metal chosen from nickel, cobalt, manganese, lithium and aluminum, $0<x<1$, and $0<z<1$.

88. The process of any one of claims 14 to 25, wherein said metal oxide is chosen from $LiNi_{0.33}Mn_{0.33}Co_{0.33}O_2$, $LiNi_{0.5}Mn_{0.3}Co_{0.2}O_2$, $LiNi_{0.6}Mn_{0.2}Co_{0.2}O_2$, $LiNi_{0.8}Mn_{0.1}Co_{0.1}O_2$ and $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$.

89. The process of any one of claims 14 to 25, wherein said lithium hydroxide obtained by said electromembrane process is used as is in aqueous composition and reacted with said obtained metal hydroxide to obtain the mixture of metal hydroxides.

90. The process of any one of claims 14 to 25, wherein said lithium hydroxide obtained by said electromembrane process is crystallized before being reacted with said obtained metal hydroxide to obtain the mixture of metal hydroxides.

91. The process of any one of claims 14 to 25, wherein said lithium hydroxide obtained by said electromembrane process is crystallized and then dissolved before being reacted with said obtained metal hydroxide to obtain the mixture of metal hydroxides.

92. The process of any one of claims 1 to 91, wherein said process comprises submitting said liquid comprising said sulfate to said electromembrane process for converting said sulfate into said hydroxide and to generate sulfuric acid.

93. The process of claim 92, wherein an anolyte of said electromembrane process is treated by substantially selectively precipitating a sulfate therefrom, thereby increasing H_2SO_4 concentration.

94. The process of claim 91, 92 or 93, wherein LiOH is substantially selectively precipitated over sodium hydroxide by evaporative crystallisation.

95. The process of claim 92, 93 or 94, comprising recovering said sulfuric acid.

96. The process of claim 92, 93, 94 or 95, comprising recovering and concentrating said sulfuric acid.

97. The process of any one of claims 1 to 96, wherein the metal sulfate is a transition metal sulfate.

98. The process of any one of claims 1 to 97, wherein the metal sulfate is obtained from a metal source.

99. The process of claim 98, wherein the metal source is a transition metal source.

100. The process of claim 98 or 99, wherein the metal sulfate is obtained by leaching or stripping the metal source.

101. The process of any one of claims 98 to 100, wherein the metal sulfate is obtained by leaching or stripping the metal source with sulfuric acid.

102. The process of any one of claims 98 to 100 as claims 98 to 100 depend on any one of claims 92 to 96, wherein the metal sulfate is obtained by leaching or stripping the metal source with said generated sulfuric acid.

103. The process of any one of claims 98 to 100, wherein the metal sulfate is obtained by leaching or stripping the metal source with electrochemically generated sulfuric acid.

104. The process of any one of claims 98 to 100, wherein the metal sulfate is obtained by leaching or stripping the metal source with electrochemically generated sulfuric acid in the presence of H_2O_2.

105. The process of any one of claims 98 to 104, wherein the metal source is a metal in its metallic form.

106. The process of any one of claims 98 to 104, wherein the metal source is nickel concentrate.

107. The process of claim 106, wherein the nickel concentrate further comprises cobalt.

108. The process of any one of claims 98 to 107, wherein the metal source is a spent battery or a constituent thereof.

109. The process of claim 108, wherein the constituent is chosen from a cathode, an anode, a black mass, slag, and mixtures thereof.

110. The process of any one of claims 98 to 109, wherein the metal sulfate is obtained by leaching the metal source.

111. The process of claim 110, wherein the metal sulfate is obtained by leaching the metal source with sulfuric acid in the presence of H_2O_2.

112. The process of any one of claims 98 to 104, wherein the metal source is an organic composition comprising nickel.

113. The process of claim 112, wherein the organic composition further comprises cobalt.

114. The process of claim 112 or 113 wherein the metal sulfate is obtained by stripping the metal source.

115. The process of any one of claims 1 to 97, wherein the metal sulfate is obtained by treating a concentrate of different metals with organic and aqueous phases so as to separate the different metals from one another and then, said metals are crystallized under the form of sulfate salts.

116. The process of any one of claims 1 to 115, wherein the reacting of the metal sulfate comprising (i) said at least one metal chosen from nickel and cobalt and optionally (ii) said at least one metal chosen from manganese, lithium and aluminum to obtain said metal hydroxide comprises reacting the metal sulfate with sodium hydroxide and lithium hydroxide, and the sodium hydroxide is used to increase pH .

117. The process of any one of claims 7, 8, and 16 to 18, wherein the reacting of the metal sulfate comprising (i) said at least one metal chosen from nickel and cobalt and optionally (ii) said at least one metal chosen from manganese, lithium and aluminum to obtain said metal hydroxide comprises reacting the metal sulfate with sodium hydroxide and lithium hydroxide.

118. The process of claim 117, wherein the sodium hydroxide is used as a pH enhancer to cause precipitation of said metal hydroxide.

119. The process of claim 1, wherein said liquid further comprises sodium sulfate.

120. The process of claim 1, wherein said liquid further comprises sodium sulfate and said liquid is submitted to said electromembrane process for converting said lithium sulfate into said lithium hydroxide and for converting said sodium sulfate into sodium hydroxide.

121. The process of claim 120, wherein the obtained lithium hydroxide is separated from said sodium hydroxide.

122. The process of claim 121, wherein said lithium hydroxide is substantially selectively precipitated over sodium hydroxide.

123. The process of claim 121, wherein said lithium hydroxide is substantially selectively precipitated over sodium hydroxide by evaporative crystallisation.

124. A process for preparing a metal oxide comprising (i) at least one metal chosen from nickel and cobalt and optionally (ii) at least one metal chosen from manganese, lithium and aluminum, said process comprising:

reacting a metal sulfate and/or a metal nitrate comprising (i) at least one metal chosen from nickel, and cobalt, and optionally (ii) at least one metal chosen from manganese, lithium and aluminum with at least one base chosen from LiOH, NaOH, KOH, RbOH, CsOH, $Mg(OH)_2$, $Ca(OH)_2$, $Sr(OH)_2$, and $Ba(OH)_2$ and optionally a chelating agent in order to obtain a solid comprising a metal hydroxide and a liquid comprising at least one of Li_2SO_4, Na_2SO_4, K_2SO_4, Rb_2SO_4, Cs_2SO_4, $MgSO_4$, $CaSO_4$, $SrSO_4$, $BaSO_4$, $LiNO_3$, $NaNO_3$, KNO_3, $RbNO_3$, $CsNO_3$, $Mg(NO_3)_2$, $Ca(NO_3)_2$, $Sr(NO_3)_2$ and $Ba(NO_3)_2$

separating said liquid and said solid from one another to obtain said metal hydroxide;

submitting said liquid comprising at least one of Li_2SO_4, Na_2SO_4, K_2SO_4, Rb_2SO_4, Cs_2SO_4, $MgSO_4$, $CaSO_4$, $SrSO_4$, $BaSO_4$, $LiNO_3$, $NaNO_3$, KNO_3, $RbNO_3$, $CsNO_3$, $Mg(NO_3)_2$, $Ca(NO_3)_2$, $Sr(NO_3)_2$ and $Ba(NO_3)_2$ to an electromembrane process for converting at least one of Li_2SO_4, Na_2SO_4, K_2SO_4, Rb_2SO_4, Cs_2SO_4, $MgSO_4$, $CaSO_4$, $SrSO_4$, $BaSO_4$, $LiNO_3$, $NaNO_3$, KNO_3, $RbNO_3$, $CsNO_3$, $Mg(NO_3)_2$, $Ca(NO_3)_2$, $Sr(NO_3)_2$ and $Ba(NO_3)_2$ into at least one of LiOH, NaOH, KOH, RbOH, CsOH, $Mg(OH)_2$, $Ca(OH)_2$, $Sr(OH)_2$, and $Ba(OH)_2$; and

reusing at least a first portion of said at least one of LiOH, NaOH, KOH, RbOH, CsOH, $Mg(OH)_2$, $Ca(OH)_2$, $Sr(OH)_2$, and $Ba(OH)_2$ obtained by said electromembrane process for reacting with said metal sulfate and/or said metal nitrate;

reacting at least a second portion of said at least one of LiOH, NaOH, KOH, RbOH, CsOH, $Mg(OH)_2$, $Ca(OH)_2$, $Sr(OH)_2$, and $Ba(OH)_2$ obtained by said electromembrane process with said obtained metal hydroxide to obtain a mixture of metal hydroxides; and

roasting said mixture of metal hydroxides to obtain said metal oxide.

125. The process of claim 124, wherein said solid is a precipitate comprising said metal hydroxide, said precipitate being obtained at a pH of about 9 to about 14.

126. The process of claim 124, wherein said solid is a precipitate comprising said metal hydroxide, said precipitate being obtained at a pH of about 10 to about 13.

127. The process of claim 124, wherein said solid is a precipitate comprising said metal hydroxide, said precipitate being obtained at a pH of about 10.5 to about 12.5.

128. The process of any one of claims 124 to 127, further comprising washing said metal hydroxide.

129. The process of claim 128, further comprising drying said metal hydroxide.

130. The process of any one of claims 124 to 129 wherein said metal sulfate is reacted with said hydroxide and a chelating agent that is chosen from ammonia, NH_4OH, acetylacetone, 5-sulfosalicylic acid, oxalic acid, EDTA (ethylenediaminetetraacetic acid), NTA (nitrilotriacetic acid), DCTA (trans-1,2-diaminocyclohexanetetraacetic acid), DTPA (diethylenetriaminepentaacetic acid), and EGTA (ethylene glycol bis(2-aminoethyl ether)-N,N,N′,N′-tetraacetic acid).

131. The process of any one of claims 124 to 130, wherein roasting said mixture of metal hydroxides comprises roasting at a first temperature of at least 350 °C for a period of time of at least 4 hours.

132. The process of any one of claims 124 to 130, wherein roasting said mixture of metal hydroxides comprises roasting at a first temperature of at least 400 °C for a period of time of at least 6 hours.

133. The process of claim 131 or 132, wherein roasting said mixture of metal hydroxides further comprises roasting at a second temperature of at least 600 °C for a period of time of at least 6 hours.

134. The process of claim 131 or 132, wherein roasting said mixture of metal hydroxides further comprises roasting at a second temperature of at least 700 °C for a period of time of at least 8 hours.

135. The process of claim 131 or 132, wherein roasting said mixture of metal hydroxides further comprises roasting at a second temperature of at least 500 °C for a period of time of at least 8 hours.

136. The process of any one of claims 124 to 135, wherein said electromembrane process comprises an electrolysis membrane process.

137. The process of any one of claims 124 to 135, wherein said electromembrane process comprises a two-compartment monopolar or bipolar membrane electrolysis process.

138. The process of claim 137, wherein said two-compartment monopolar or bipolar membrane electrolysis process is carried out in a first electrochemical cell comprising an anolyte compartment separated from a catholyte compartment by a cation exchange membrane.

139. The process of claim 138, wherein said cation exchange membrane comprises a perfluorinated sulfonic acid.

140. The process of claim 138 or 139 wherein during said two-compartment monopolar or bipolar membrane electrolysis process, an aqueous stream comprising said liquid comprising at least one of Li_2SO_4, Na_2SO_4, K_2SO_4, Rb_2SO_4, Cs_2SO_4, $MgSO_4$, $CaSO_4$, $SrSO_4$, $BaSO_4$, $LiNO_3$, $NaNO_3$, KNO_3, $RbNO_3$, $CsNO_3$, $Mg(NO_3)_2$, $Ca(NO_3)_2$, $Sr(NO_3)_2$ and $Ba(NO_3)_2$ is introduced into said anolyte compartment, a first metal-reduced aqueous stream is removed from said anolyte compartment and a first lithium hydroxide-enriched aqueous stream is removed from said catholyte compartment.

141. The process of claim 140, wherein during said two-compartment monopolar or bipolar membrane electrolysis process, conversion of said liquid comprising at least one of Li_2SO_4, Na_2SO_4, K_2SO_4, Rb_2SO_4, Cs_2SO_4, $MgSO_4$, $CaSO_4$, $SrSO_4$, $BaSO_4$, $LiNO_3$, $NaNO_3$, KNO_3, $RbNO_3$, $CsNO_3$, $Mg(NO_3)_2$, $Ca(NO_3)_2$, $Sr(NO_3)_2$ and $Ba(NO_3)_2$ to at least one of LiOH, NaOH, KOH, RbOH, CsOH, $Mg(OH)_2$, $Ca(OH)_2$, $Sr(OH)_2$, and

$Ba(OH)_2$ proceeds until hydroxide current efficiency is no longer at least substantially maintained so that it decreases.

142. The process of claim 140, wherein said liquid comprises Li_2SO_4 and the Li_2SO_4 is converted to LiOH, and wherein during said two-compartment monopolar or bipolar membrane electrolysis process, conversion of said lithium sulfate to lithium hydroxide proceeds until pH in said anolyte compartment is a value of from about 0.4 to about 1.0.

143. The process of claim 140, wherein said liquid comprises Li_2SO_4 and the Li_2SO_4 is converted to LiOH, and wherein during said two-compartment monopolar or bipolar membrane electrolysis process, conversion of said lithium sulfate to lithium hydroxide proceeds until pH in said anolyte compartment is a value of about 0.5 to about 0.7.

144. The process of any one of claims 140 to 143, wherein said liquid comprises Li_2SO_4 and the Li_2SO_4 is converted to LiOH, and wherein in said catholyte compartment of said two-compartment monopolar or bipolar membrane electrolysis process, lithium hydroxide is at least substantially maintained at a concentration of about 1 M to about 4 M.

145. The process of any one of claims 140 to 143, wherein said liquid comprises Li_2SO_4 and the Li_2SO_4 is converted to LiOH, and wherein in said catholyte compartment of said two-compartment monopolar or bipolar membrane electrolysis process, lithium hydroxide is at least substantially maintained at a concentration of about 2 M to about 3 M.

146. The process of any one of claims 140 to 145, wherein during said two-compartment monopolar or bipolar membrane electrolysis process, said aqueous stream comprising lithium sulfate is introduced into said anolyte compartment at a temperature of about 20 °C to about 100 °C.

147. The process of any one of claims 140 to 145, wherein during said two-compartment monopolar or bipolar membrane electrolysis process, said aqueous stream comprising lithium sulfate is introduced into said anolyte compartment at a temperature of about 40 °C to about 70 °C.

148. The process of any one of claims 140 to 145, wherein during said two-compartment monopolar or bipolar membrane electrolysis process, said aqueous stream comprising lithium sulfate is introduced into said anolyte compartment at a temperature of about 45 °C to about 55 °C.

149. The process of any one of claims 140 to 145, wherein during said two-compartment monopolar or bipolar membrane electrolysis process, said aqueous stream comprising lithium sulfate and is introduced into said anolyte compartment at a temperature of about 50°C.

150. The process of any one of claims 142 to 145, wherein during said two-compartment monopolar or bipolar membrane electrolysis process, said first metal-reduced aqueous stream is a first lithium-reduced stream, and said first lithium-reduced aqueous stream is removed from said anolyte compartment at a temperature of about 50 °C to about 85 °C.

151. The process of any one of claims 140 to 145, wherein during said two-compartment monopolar or bipolar membrane electrolysis process, said aqueous stream comprising lithium sulfate is introduced into said anolyte compartment at a temperature of about 55 °C to about 65 °C.

152. The process of any one of claims 142 to 145, wherein during said two-compartment monopolar or bipolar membrane electrolysis process, said first metal-reduced aqueous stream is a first lithium-reduced stream, and said first lithium-reduced aqueous stream is removed from said anolyte compartment at a temperature of about 60°C.

153. The process of any one of claims 140 to 145, wherein during said two-compartment monopolar or bipolar membrane electrolysis process, temperature in said first electrochemical cell is at least substantially maintained at a value of about 50 °C to about 85 °C.

154. The process of any one of claims 140 to 145, wherein in said two-compartment monopolar or bipolar membrane electrolysis process, current density is at least substantially maintained at a value of from about 0.5 kA/m^2 to about 6 kA/m^2.

155. The process of any one of claims 140 to 145, wherein in said two-compartment monopolar or bipolar membrane electrolysis process, current density is at least substantially maintained at a value of from about 3 kA/m^2 to about 5 kA/m^2.

156. The process of any one of claims 140 to 145, wherein in said two-compartment monopolar or bipolar membrane electrolysis process, voltage is at least substantially maintained at a value of about 3 V to about 8 V.

157. The process of any one of claims 140 to 145, wherein in said two-compartment monopolar or bipolar membrane electrolysis process, voltage is at least substantially maintained at a value of from about 5 V to about 10 V.

158. The process of any one of claims 140 to 157, wherein said first electrochemical cell has a cell area of about 0.2 m^2 to about 4 m^2.

159. The process of any one of claims 140 to 157, wherein said first electrochemical cell has a cell area of about 0.5 m^2 to about 3.5 m^2.

160. The process of any one of claims 140 to 157, wherein said first electrochemical cell has a cell area of about 1 m^2 to about 3 m^2.

161. The process of any one of claims 140 to 157, wherein said first electrochemical cell has a cell area of about 1 m^2 to about 2 m^2.

162. The process of any one of claims 124 to 135, wherein said electromembrane process comprises an electrodialysis process.

163. The process of any one of claims 124 to 135, wherein said electromembrane process comprises a three-compartment monopolar or bipolar membrane electrolysis process.

164. The process of claim 163, wherein said three-compartment monopolar or bipolar membrane electrolysis process is carried out in a second electrochemical cell comprising an anolyte compartment separated from a central compartment by an anion exchange membrane and a catholyte

compartment separated from said central compartment by a cation exchange membrane.

165. The process of claim 164, wherein said cation exchange membrane comprises a sulfonated polytetrafluoroethylene.

166. The process of claim 164 or 165, wherein during said three-compartment monopolar or bipolar membrane electrolysis process, a first lithium-reduced aqueous stream is introduced into said central compartment, a second lithium-reduced aqueous stream is removed from said central compartment and a second lithium hydroxide-enriched aqueous stream is removed from said catholyte compartment.

167. The process of claim 166, wherein said three-compartment monopolar or bipolar membrane electrolysis process further comprises producing sulfuric acid in said anolyte compartment and removing a sulfuric acid-containing aqueous stream from said anolyte compartment.

168. The process of claim 167, wherein in said three-compartment monopolar or bipolar membrane electrolysis process, said anion exchange membrane is a proton-blocking membrane.

169. The process of claim 167 or 168, wherein in said anolyte compartment of said three-compartment monopolar or bipolar membrane electrolysis process, said sulfuric acid is at least substantially maintained at a concentration of sulfuric acid of about 0.1 M to about 2 M.

170. The process of claim 167 or 168, wherein in said anolyte compartment of said three-compartment monopolar or bipolar membrane electrolysis process, said sulfuric acid is at least substantially maintained at a concentration of sulfuric acid of about 0.7 M to about 1.2 M.

171. The process of any one of claims 166 to 168, wherein in said catholyte compartment of said three-compartment membrane electrolysis process, said lithium hydroxide is at least substantially maintained at a concentration of about 1 M to about 4 M.

172. The process of any one of claims 166 to 168, wherein in said catholyte compartment of said three-compartment membrane electrolysis process, said lithium hydroxide is at least substantially maintained at a concentration of about 2 M to about 3 M.

173. The process of any one of claims 166 to 172, wherein during said three-compartment monopolar or bipolar membrane electrolysis process, said first lithium-reduced aqueous stream is introduced into said central compartment at a temperature of about 40 °C to about 85 °C.

174. The process of any one of claims 166 to 172, wherein during said three-compartment monopolar or bipolar membrane electrolysis process, said second lithium-reduced aqueous stream is removed from said central compartment at a temperature of about 40 °C to about 80 °C.

175. The process of any one of claims 166 to 172, wherein during said three-compartment monopolar or bipolar membrane electrolysis process, temperature in said second electrochemical cell is at least substantially maintained at a value of about 50°C to about 70°C.

176. The process of any one of claims 166 to 175, wherein in said three-compartment monopolar or bipolar membrane electrolysis process, current density is at least substantially maintained at a value of about 0.5 kA/m^2 to about 5 kA/m^2.

177. The process of any one of claims 166 to 175, wherein in said three-compartment monopolar or bipolar membrane electrolysis process, current density is at least substantially maintained at a value of about 1 kA/m^2 to about of about 10 kA/m^2.

178. The process of any one of claims 166 to 175, wherein in said three-compartment monopolar or bipolar membrane electrolysis process, voltage is at least substantially maintained at a value of about 5 V to about 9 V.

179. The process of any one of claims 166 to 178, wherein said electrochemical cell has a cell area of about 0.2 m^2 to about 4 m^2.

180. The process of any one of claims 166 to 178, wherein said electrochemical cell has a cell area of about 0.5 m^2 to about 3.5 m^2.

181. The process of any one of claims 166 to 178, wherein said electrochemical cell has a cell area of about 1 m^2 to about 3 m^2.

182. The process of any one of claims 166 to 178, wherein said electrochemical cell has a cell area of about 1 m^2 to about 2 m^2.

183. The process of claim 166, wherein said three-compartment monopolar or bipolar membrane electrolysis process further comprises introducing ammonia into said anolyte compartment, producing ammonium sulfate in said anolyte compartment and removing an ammonium sulfate-containing aqueous stream from said anolyte compartment.

184. The process of claim 183, wherein in said three-compartment monopolar or bipolar membrane electrolysis process, said anion exchange membrane is not a proton-blocking membrane.

185. The process of claim 184, wherein said anion exchange membrane is an Astom^{TM} AHA membrane.

186. The process of any one of claims 183 to 185, wherein in said anolyte compartment of said three-compartment monopolar or bipolar membrane electrolysis process, said ammonium sulfate is at least substantially maintained at a concentration of ammonium sulfate of about 0.5 M to about 4 M.

187. The process of any one of claims 183 to 185, wherein in said anolyte compartment of said three-compartment monopolar or bipolar membrane electrolysis process, said ammonium sulfate is at least substantially maintained at a concentration of ammonium sulfate of about 3 M.

188. The process of any one of claims 183 to 187, wherein in said catholyte compartment of said three-compartment monopolar or bipolar membrane electrolysis process, said lithium hydroxide is at least substantially maintained at a concentration of about 1 M to about 3 M.

189. The process of any one of claims 183 to 187, wherein in said catholyte compartment of said three-compartment monopolar or bipolar membrane electrolysis process, said lithium hydroxide is at least substantially maintained at a concentration of about 2 M.

190. The process of any one of claims 124 to 135, wherein said electromembrane process is electrolysis.

191. The process of any one of claims 124 to 135, wherein said electromembrane process is electrodialysis.

192. The process of any one of claims 124 to 191, wherein the chelating agent is chosen from NH_3, NH_4OH, acetylacetone, 5-sulfosalicylic acid, and oxalic acid.

193. The process of any one of claims 124 to 191, wherein the chelating agent is chosen from EDTA (ethylenediaminetetraacetic acid), NTA (nitrilotriacetic acid), DCTA (trans-1,2-diaminocyclohexanetetraacetic acid), DTPA (diethylenetriaminepentaacetic acid), and EGTA (ethylene glycol bis(2-aminoethyl ether)-N,N,N′,N′-tetraacetic acid).

194. The process of claim 193, wherein said chelating agent is present.

195. The process of any one of claims 124 to 194, wherein said lithium hydroxide obtained by said electromembrane process is used as is in aqueous composition and reacted with said obtained metal hydroxide to obtain the mixture of metal hydroxides.

196. The process of any one of claims 124 to 194, wherein said lithium hydroxide obtained by said electromembrane process is crystallized before being reacted with said obtained metal hydroxide to obtain the mixture of metal hydroxides.

197. The process of any one of claims 124 to 194, wherein said lithium hydroxide obtained by said electromembrane process is crystallized and then dissolved before being reacted with said obtained metal hydroxide to obtain the mixture of metal hydroxides.

198. The process of any one of claims 124 to 197, wherein said process comprises submitting said liquid comprising lithium sulfate to said electromembrane process for converting said lithium sulfate into lithium hydroxide and to generate sulfuric acid.

199. The process of claim 198, comprising recovering said sulfuric acid.

200. The process of claim 198, comprising recovering and concentrating said sulfuric acid.

201. The process of any one of claims 124 to 200, wherein the metal sulfate is a sulfate of at least one metal chosen from nickel and cobalt, and optionally manganese.

202. The process of any one of claims 124 to 201, wherein the metal sulfate is obtained from a metal source.

203. The process of claim 202, wherein the metal source is a transition metal source.

204. The process of claim 202 or 203, wherein the metal sulfate is obtained by leaching or stripping the metal source.

205. The process of any one of claims 202 to 204, wherein the metal sulfate is obtained by leaching or stripping the metal source with sulfuric acid.

206. The process of any one of claims 202 to 204 as claims 202 to 204 depend on any one of claims 198 to 200, wherein the metal sulfate is obtained by leaching or stripping the metal source with said generated sulfuric acid.

207. The process of any one of claims 202 to 204, wherein the metal sulfate is obtained by leaching or stripping the metal source with electrochemically generated sulfuric acid.

208. The process of any one of claims 202 to 207, wherein the metal source is a metal in its metallic form.

209. The process of any one of claims 202 to 207, wherein the metal source is nickel concentrate.

210. The process of claim 209, wherein the nickel concentrate further comprises cobalt.

211. The process of any one of claims 202 to 207, wherein the metal source is a spent battery or a constituent thereof.

212. The process of claim 211, wherein the constituent is chosen from a cathode, an anode, a black mass, slag, and mixtures thereof.

213. The process of any one of claims 202 to 212, wherein the metal sulfate is obtained by leaching the metal source.

214. The process of any one of claims 202 to 207, wherein the metal source is an organic composition comprising nickel.

215. The process of claim 214, wherein the organic composition further comprises cobalt.

216. The process of claim 214 or 215, wherein the metal sulfate is obtained by stripping the metal source.

217. The process of any one of claims 124 to 216, wherein sodium hydroxide is used to increase pH and reacted with (a) said metal sulfate comprising (i) said at least one metal chosen from nickel and cobalt and optionally (ii) said at least one metal chosen from manganese, lithium and aluminum; and (b) said lithium hydroxide, to obtain said metal hydroxide.

218. The process of any one of claims 124 to 216, wherein sodium hydroxide is reacted with said metal sulfate and lithium hydroxide.

219. The process of claim 125 to 127, wherein sodium hydroxide is used as a pH enhancer to cause precipitation of said metal hydroxide.

220. The process of any one of claims 124 to 219, wherein said liquid further comprises sodium sulfate.

221. The process of any one of claims 124 to 219, wherein said liquid further comprises sodium sulfate and said liquid is submitted to said electromembrane process for converting said lithium sulfate into said lithium hydroxide and for converting said sodium sulfate into sodium hydroxide.

222. The process of claim 221, wherein the obtained lithium hydroxide is separated from said sodium hydroxide.

223. The process of claim 222, wherein said lithium hydroxide is substantially selectively precipitated over sodium hydroxide.

224. The process of claim 222, wherein said lithium hydroxide is substantially selectively precipitated over sodium hydroxide by evaporative crystallisation.

225. The process of claim 1 or 2, wherein the obtained metal hydroxide is reacted with an aqueous composition comprising lithium hydroxide to obtain a hydroxides composition, said hydroxides composition being then dried to obtain a mixture of hydroxides.

226. The process of claim 225, wherein said hydroxides composition is dried by means of a spray dryer.

227. The process of any one of claims 1 to 123, wherein the metal sulfate or metal hydroxide is purified by solvent extraction and wherein the solvent is chosen from phosphorous acid, hydroxy-oxime extractants, and β-diketone metal cation extractants.

228. The process of claim 227, wherein the phosphorous acid is chosen from Cyanex™ 272, Cyanex™ 301, Cyanex™ 302, Di-(2-ethylhexyl)phosphoric acid (D2EHPA), DEHTPA, Baysolvex™ DEDP, Ionquest™ 801, Hoe F 3787 ™, MEHPA, P204 ™, PC88A ™, and P507 ™.

229. The process of claim 227, wherein the hydroxyl-oxime extractants are chosen from Acorga™ P50, Acorga™ K2000, LIX™ 84-I, SME™ 529, LIX™ 65N, LIX™ 64, LIX™ 70, LIX™ 860, and LIX™ 622.

230. The process of claim 227, wherein the β-diketone metal cation extractants are chosen from LIX™ 54, XI-N54™, XI-55 ™, and XI-57 ™.

231. The process of any one of claims 1 to 123, wherein the reacting of the metal sulfate to obtain said solid comprising said metal hydroxide is further with an addition of NH_4OH.

232. The process of claim 108 or 109, wherein the metal sulfate is obtained by leaching the spent battery and the leaching of the spent battery is done with a leaching solution chosen from at least one of H_2SO_4, H_2O_2, HNO_3, HCl, nitric acid, citric acid, oxalic acid, aspartic acid, ascorbic acid, and glucose.

233. The process of any one of claims 14 to 25, wherein said roasting is carried out with agitation so as to ensure completion of the conversion into said metal oxide.

234. The process of any one of claims 14 to 84, wherein said liquid is separated from said solid by filtration, and wherein said filtered liquid is optionally purified and/or concentrated before entering the electromembrane process.

235. The process of any one of claims 100 to 104, 110, 111, 204 to 207, and 213, wherein the leaching produces a leached solution, and the leached solution is purified before the reacting of the metal sulfate.

236. The process of any one of claims 1 to 226 and 233 to 235, wherein the metal sulfate and/or the metal hydroxide is purified by a process chosen from metals selective separation, precipitation of hydroxides, precipitation of insoluble salts, oxidative precipitation, ion exchange, solvent extraction, electrochemical plating and crystallization.

237. The process of any one of claims 1 to 226 and 233 to 235, wherein the metal sulfate and/or the metal hydroxide is purified and the purification of the metal sulfate and/or the metal hydroxide is carried out by selective precipitation by addition of O_2, SO_2, H_2SO_5, persulfates, $(NH_4)_2C_2O_4$,

chlorine, chlorine compounds, O_3, NaOCl, CoS, Na_2S, NaHS, $CaCO_3$ or Na_3PO_4.

238. The process of claim 237, wherein the persulfate is $(NH_4)_2S_2O_8$.

239. The process of claim 237, wherein the chlorine compounds are chosen from HCl, ClO_2, and $HClO_3$.

240. The process of any one of claims 1 to 226 and 233 to 235, wherein the metal sulfate and/or the metal hydroxide is purified and the purification of the metal hydroxide is carried out by precipitation of hydroxides by addition of any one of LiOH, NaOH and NH_4OH.

241. The process of any one of claims 1 to 226 and 233 to 235, wherein the metal sulfate and/or the metal hydroxide is purified and the purification of the metal hydroxide and/or the metal sulfate is carried out by addition of dimethylglyoxime.

242. The process of any one of claims 100 to 104, 110, 111, 204 to 207, 213, 232, and 235, wherein the solid/liquid (gram of material / volume of liquid) ratio in g/L for the leaching step comprises between 1/5 to 1:100.

243. The process of claim 100 or 204, wherein the leaching is carried out with at least one of H_2SO_4, H_2O_2, HNO_3, HCl, nitric acid, citric acid, oxalic acid, aspartic acid, ascorbic acid and glucose.

244. The process of any one of claims 1 to 235, wherein the metal sulfate $M(SO_4)$ and/or $Al_2(SO_4)_3$ are optionally crystallized before being reacted to obtain said solid comprising said metal hydroxide, and wherein M is chosen from at least one of Ni, Co, and Mn.

245. The process of claim 108, wherein a $LiPF_6$ electrolyte is recovered.

246. The process of any one of claims 15 to 51 and 124 to 216, wherein the metal oxide is a core-shell material with a concentration gradient from the core to the surface for the different metals, as $[LiNi_xM1_yM2_zO_2]_{core}/[LiNi_aM1_bM2_cO_2]_{shell}$, with x+y+z = 1, a+b+c = 1, M1

= Mn, Co or Al and M2 = Mn, Co or Al, and $a \neq x$ for Ni being different, leading to the concentration gradient in the metal oxide.

247. The process of any one of claims 15 to 51 and 124 to 216, wherein the metal oxide is chosen from $LiNi_{0.33}Mn_{0.33}Co_{0.33}O_2$, $LiNi_{0.5}Mn_{0.3}Co_{0.2}O_2$, $LiNi_{0.6}Mn_{0.2}Co_{0.2}O_2$, $LiNi_{0.8}Mn_{0.1}Co_{0.1}O_2$ and $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$, or $[LiNi_xM1_yM2_zO_2]_{core}/[LiNi_aM1_bM2_cO_2]_{shell}$, with M1 = Mn, Co or Al and M2 = Mn, Co or Al with x+y+z = 1, a+b+c = 1.

248. The process of any one of claims 15 to 51 and 124 to 194, wherein the metal oxide is of formula $LiMO_2$, or $Li(1+x)M(1-x)O_2$ for lithium-rich and $Li_{(1-z)}M_{(1+z)}O_2$ for Li-deficient, wherein M is at least one metal chosen from nickel and cobalt, and optionally at least one metal chosen from manganese, lithium and aluminum, 0<x<1, and 0<z<1.

249. The process of any one of claims 15 to 51 and 124 to 216, wherein the metal oxide is of formula $Li[Ni_dM1_eM2_f]O_2$ with d+e+f = 1, being made of a core $[LiNi_xM1_yM2_zO_2]$ with x+y+z = 1 and a shell $[LiNi_aM1_bM2_cO_2]$ with a+b+c = 1, with M1 = Mn, Co or Al and M2 = Mn, Co or Al and with $x<d<a$, $y<e<b$, $z<f<c$.

250. The process of any one of claims 124 to 220 and 246 to 249, wherein said metal hydroxide is $NiCoAl(OH)_2$ or $NiMnCo(OH)_2$.

251. The process of any one of claims 124 to 220 and 246 to 249, wherein said metal hydroxide is chosen from $Ni_{0.8}Co_{0.15}Al_{0.05}(OH)_2$, $Ni_{0.8}Mn_{0.1}Co_{0.1}(OH)_2$ and $Ni_{0.6}Mn_{0.2}Co_{0.2}(OH)_2$.

252. The process of any one of claims 124 to 220 and 246 to 249, wherein said metal oxide is of formula $LiMO_2$, wherein M comprises at least one of metal chosen from nickel and cobalt, and optionally at least one metal chosen from manganese, lithium and aluminum.

253. The process of any one of claims 124 to 220 and 246 to 249, wherein said metal oxide is chosen from $LiNi_{0.33}Mn_{0.33}Co_{0.33}O_2$,

$LiNi_{0.5}Mn_{0.3}Co_{0.2}O_2$, $LiNi_{0.6}Mn_{0.2}Co_{0.2}O_2$, $LiNi_{0.8}Mn_{0.1}Co_{0.1}O_2$ and $LiNi_{0.8}Co_{0.15}Al_{0.05}O_2$.

254. The process of any one of claims 202 to 209, wherein the metal source is at least substantially pure metal leached by an electrochemically generated sulfuric acid, optionally, when claims 206 to 209 refers to any one of claims 198 to 200, the metal source is at least substantially pure metal leached by the generated sulfuric acid as defined in any one of claims 198 to 200.

255. The process of any one of claims 206 to 207, and 214, wherein the metal source is a nickel concentrate that optionally comprises cobalt, optionally leached by an electrochemically generated sulfuric acid, optionally when claims 206 to 207, and 214 refer to any one of claims 198 to 200, the metal source is leached by the generated sulfuric acid as defined in any one of claims 198 to 200.

256. The process of any one of claims 202 to 210, wherein the metal source is a nickel-containing material or a cobalt-containing material.

257. The process of claim 256, wherein the metal source is chosen from nickel oxide ore, nickel matte, nickel sulfide, mixed sulfide of nickel and cobalt, crude nickel sulfate, and nickel oxide.

258. The process of claim 257, wherein the crude nickel sulfate is produced from a copper smelting process.

259. The process of claim 257, wherein the nickel oxide is leached by electrochemically generated sulfuric acid.

260. The process of any one of claims 202 to 206, wherein the metal source is an aqueous nickel-cobalt solution produced by leaching the metal source with an electrochemically generated sulfuric acid.

261. The process of any one of claims 202 to 206, wherein the metal source is an organic solution containing nickel and optionally cobalt that can optionally be stripped by an electrochemically generated sulfuric acid,

optionally when claims 202 to 206 refers to any one of claims 198 to 200, the metal source is stripped by the generated sulfuric acid as defined in any one of claims 198 to 200.

262. The process of any one of claims 202 to 206, wherein the metal source is a spent battery leached or constituent thereof leached by an electrochemically generated sulfuric acid, optionally, when claims 202 to 206 refers to any one of claims 198 to 200, the spent battery or constituent thereof is leached by the generated sulfuric acid.

263. The process of claim 262, wherein the spent battery or constituent thereof is chosen from a cathode, an anode, black mass, slag, and mixtures thereof.

264. The process of claim 263, wherein the spent battery or constituent thereof is the cathode.

265. The process of claim 263, wherein the spent battery or constituent thereof is the cathode and the anode.

266. The process of claim 263, wherein the spent battery or constituent thereof is the black mass.

267. The process of any one of claims 1 to 13, wherein the metal hydroxide is chosen from nickel-cobalt-manganese hydroxides, nickel-cobalt-aluminum hydroxides, lithium-cobalt hydroxides, nickel hydroxides, nickel-cobalt-manganese oxyhydroxides, nickel-cobalt-aluminum oxyhydroxides, nickel oxyhydroxides and lithium-cobalt oxyhydroxides.

268. The process of any one of claims 14 to 51, and 124 to 194, wherein the metal oxide is chosen from lithium-nickel-cobalt-manganese oxides, lithium nickel-cobalt-aluminum oxides, lithium nickel oxide and lithium-cobalt oxides.

269. The process of any one of claims 1 to 268, wherein an anolyte generated during the electromembrane process is treated to recover a sulfate and said sulfate is mixed with a sulfate obtained from the reacting of the

metal sulfate, said sulfates being mixed together and recycled to the electromembrane process.

270. The process of any one of claims 124 to 222, wherein H_2SO_4 generated during the electromembrane process is separated from lithium sulfate, sodium sulfate and/or potassium sulfate through anolyte concentration.

271. The process of any one of claims 124 to 222, wherein H_2SO_4 generated during the electromembrane process is separated from lithium sulfate, sodium sulfate and/or potassium sulfate through anolyte concentration by evaporative crystallization.

272. The process of any one of claims 124 to 222, wherein H_2SO_4 generated during the electromembrane process is separated from lithium sulfate, sodium sulfate and/or potassium sulfate through anolyte concentration by selective crystallization of a sulfate monohydrate.

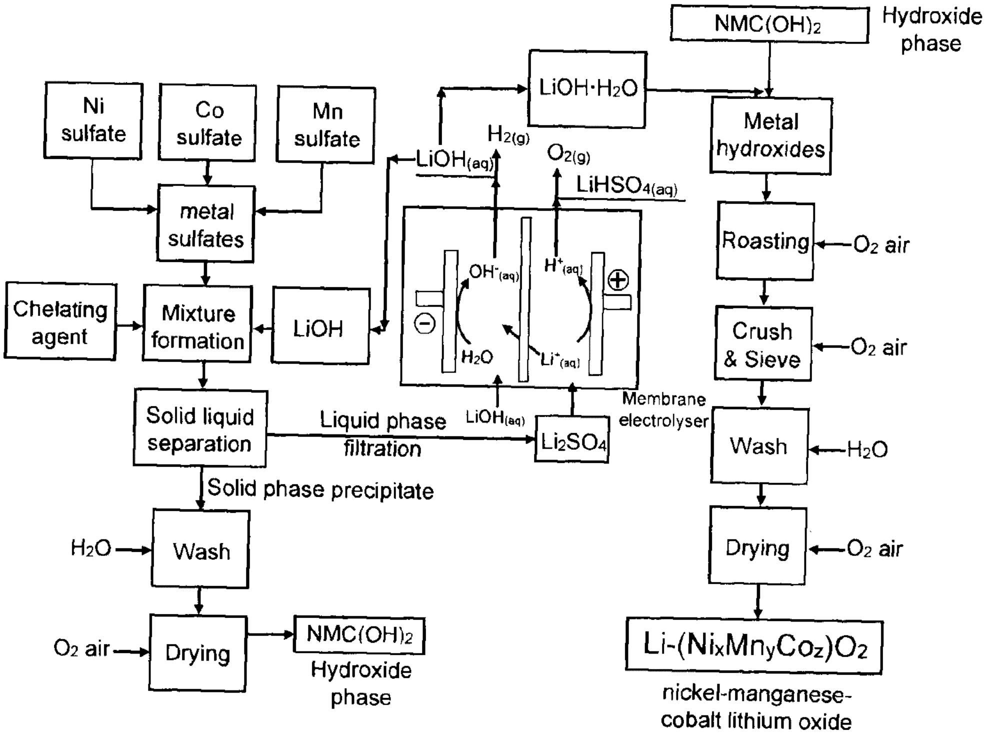

Ni sulfate
Co sulfate
Mn sulfate
metal sulfates
Chelating agent
Mixture formation
LiOH
Solid liquid separation
Liquid phase filtration
Solid phase precipitate
H_2O
Wash
O_2 air
Drying
$NMC(OH)_2$
Hydroxide phase
Li_2SO_4
$LiOH_{(aq)}$
Membrane electrolyser
$H_{2(g)}$
$O_{2(g)}$
$LiHSO_{4(aq)}$
$OH^-_{(aq)}$
$H^+_{(aq)}$
$Li^+_{(aq)}$
H_2O
$LiOH \cdot H_2O$
$NMC(OH)_2$
Hydroxide phase
Metal hydroxides
Roasting
O_2 air
Crush & Sieve
O_2 air
Wash
H_2O
Drying
O_2 air
$Li-(Ni_xMn_yCo_z)O_2$
nickel-manganese-cobalt lithium oxide

FIG. 1

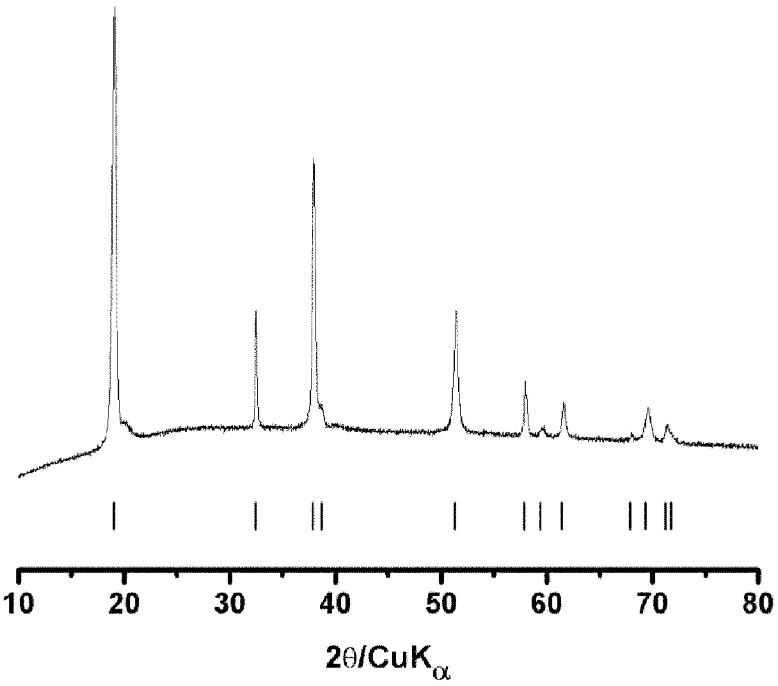


FIG. 2

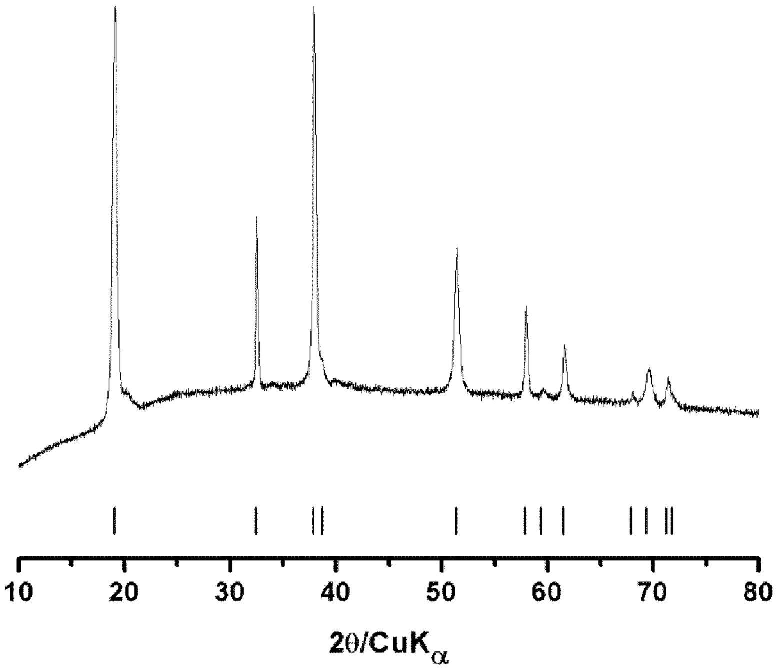


FIG. 3

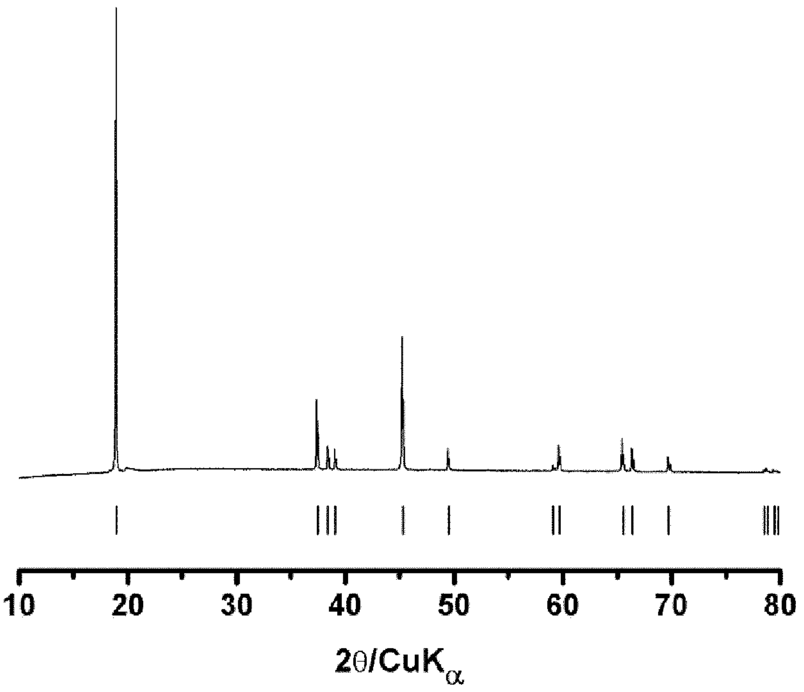


FIG. 4

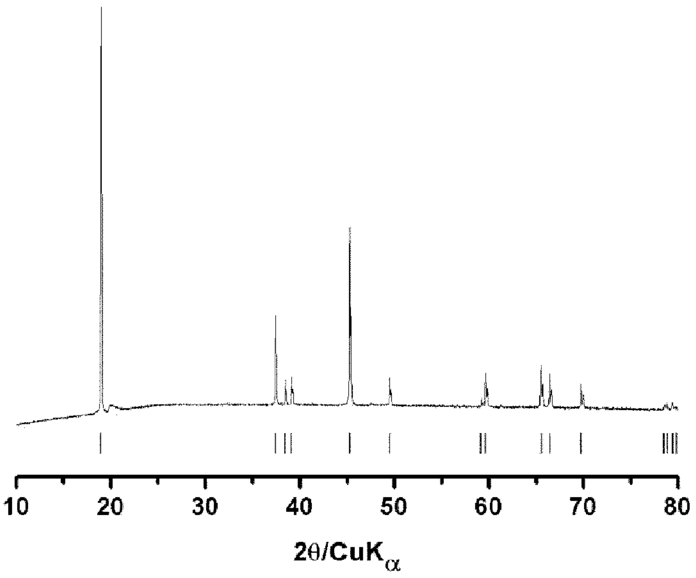
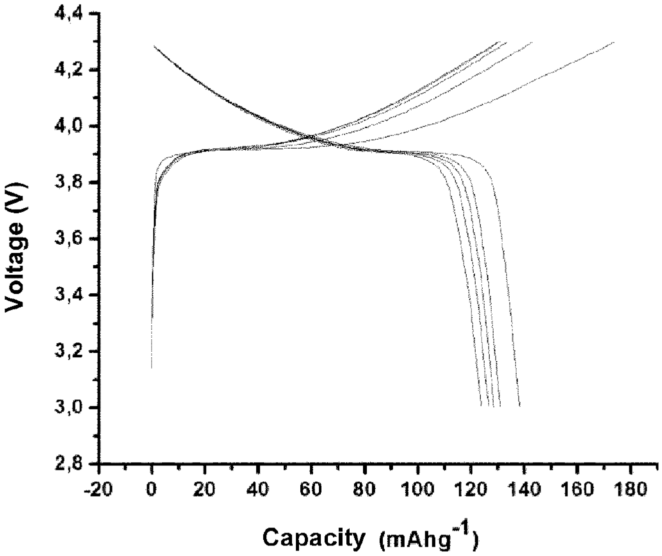


FIG. 5



Voltage (V)
4,4
4,2
4,0
3,8
3,6
3,4
3,2
3,0
2,8
-20
0
20
40
60
80
100
120
140
160
180
Capacity (mAhg^{-1})

FIG. 6

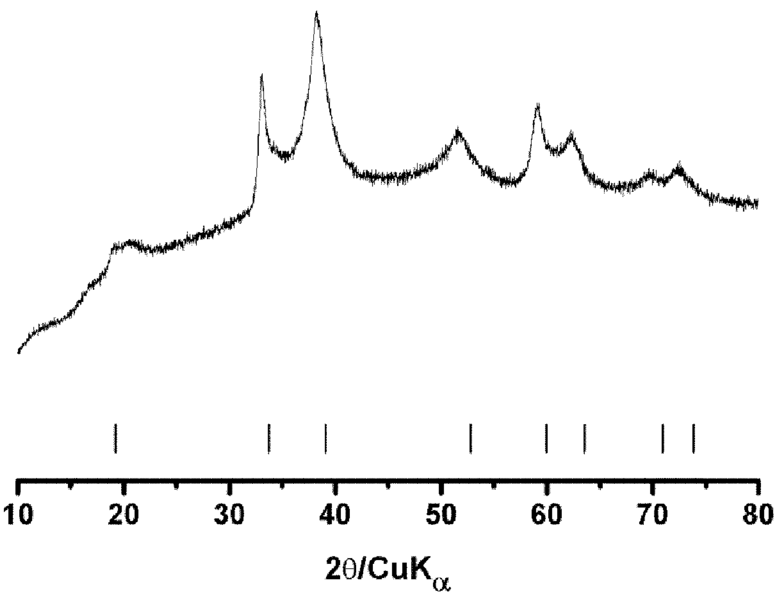


FIG. 7

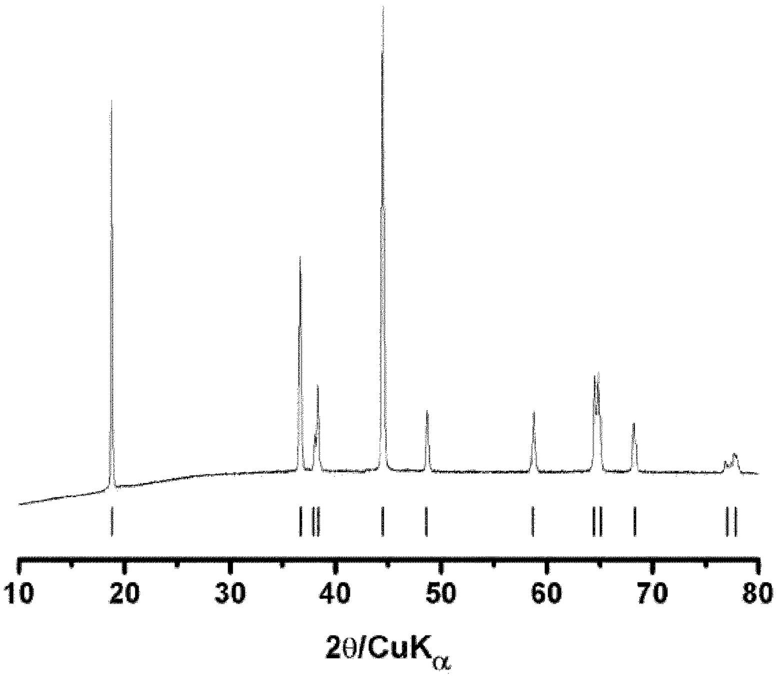


FIG. 8

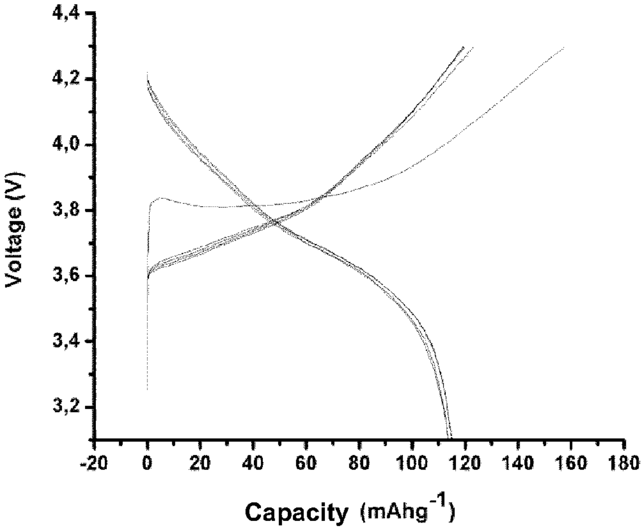

4,4
4,2
4,0
3,8
3,6
3,4
3,2
Voltage (V)
-20
0
20
40
60
80
100
120
140
160
180
Capacity (mAhg^{-1})

FIG. 9

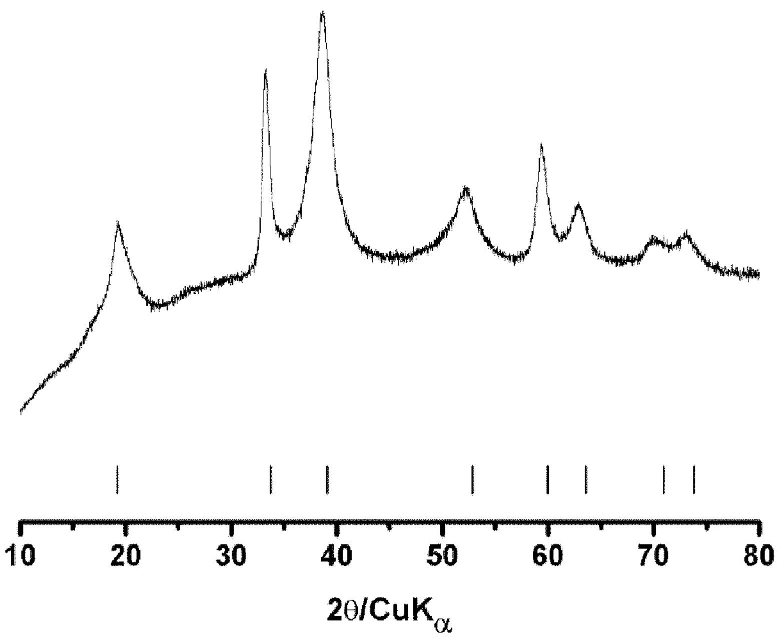


FIG. 10

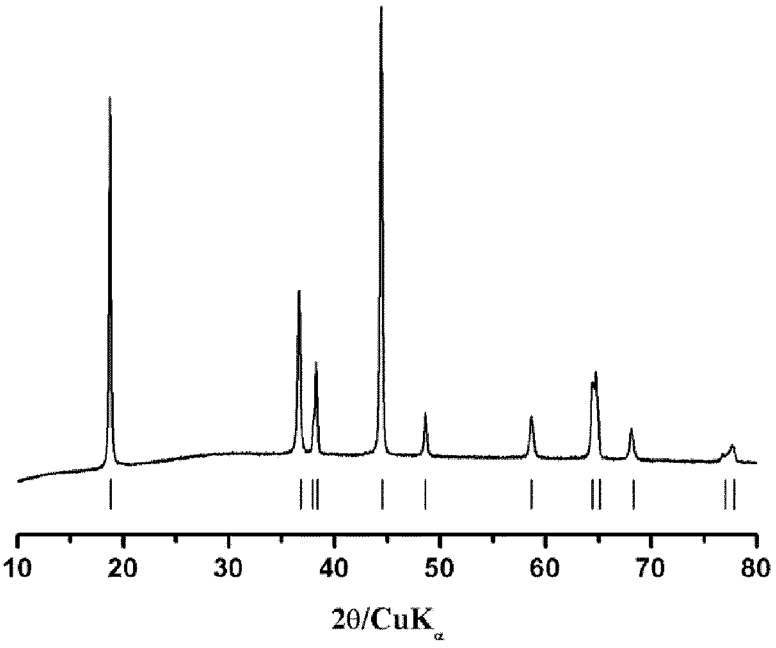


FIG. 11

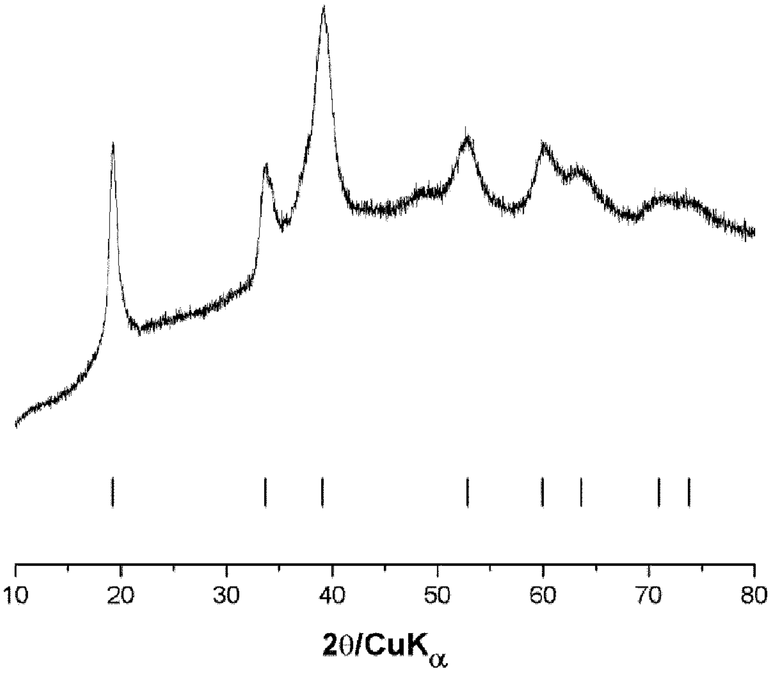


FIG. 12

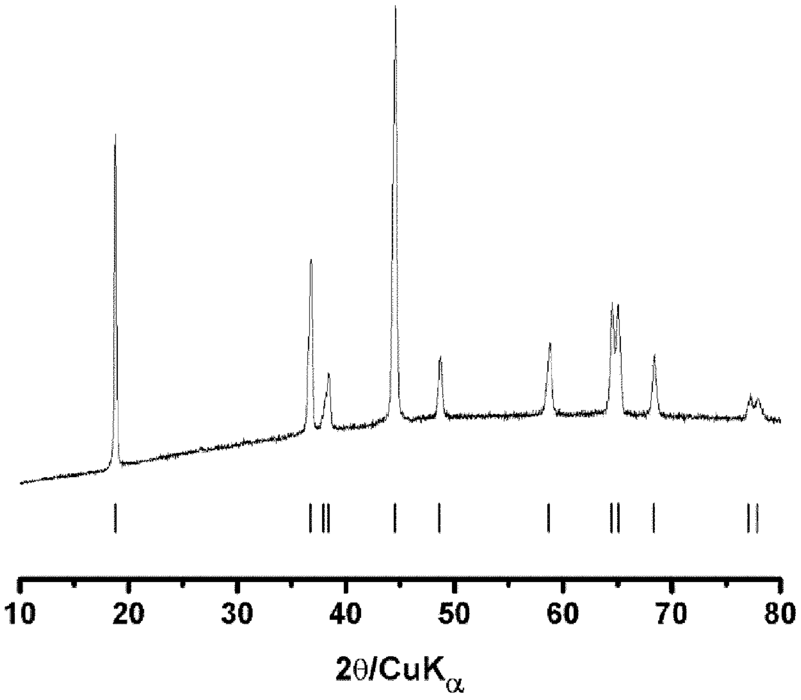
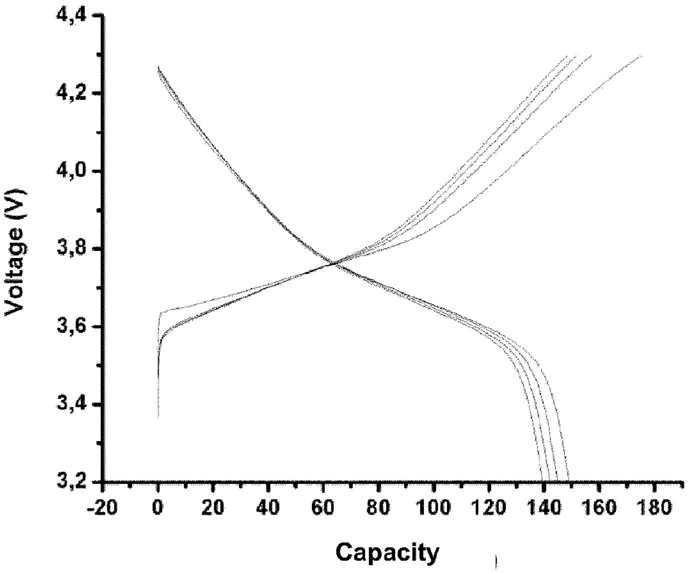


FIG. 13



4,4
4,2
4,0
3,8
3,6
3,4
3,2
Voltage (V)
-20
0
20
40
60
80
100
120
140
160
180
Capacity

FIG. 14

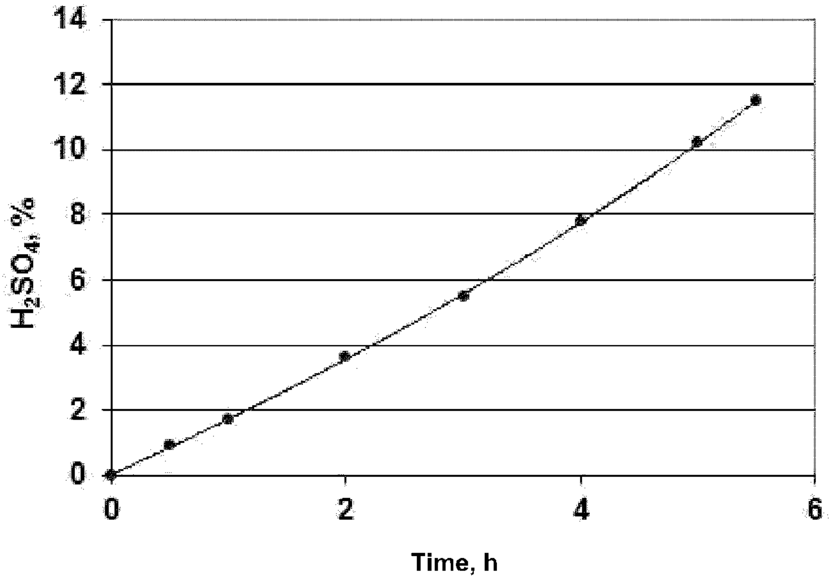


FIG. 15

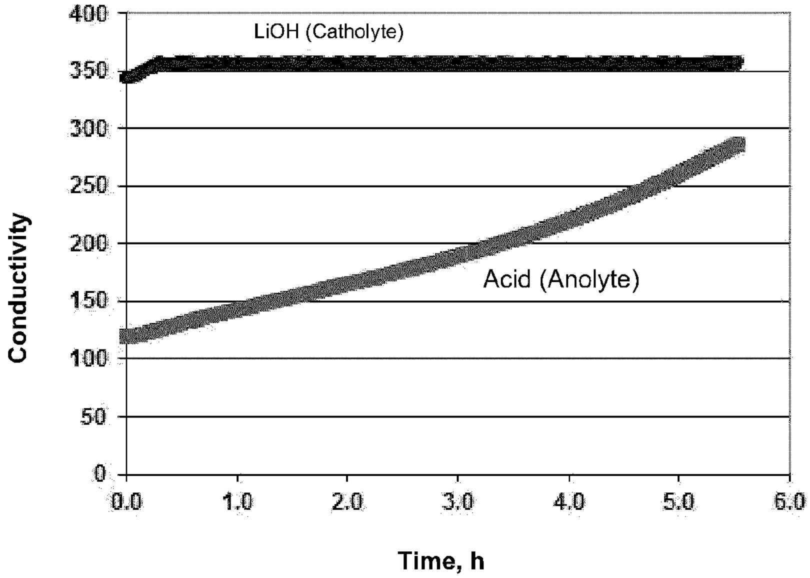
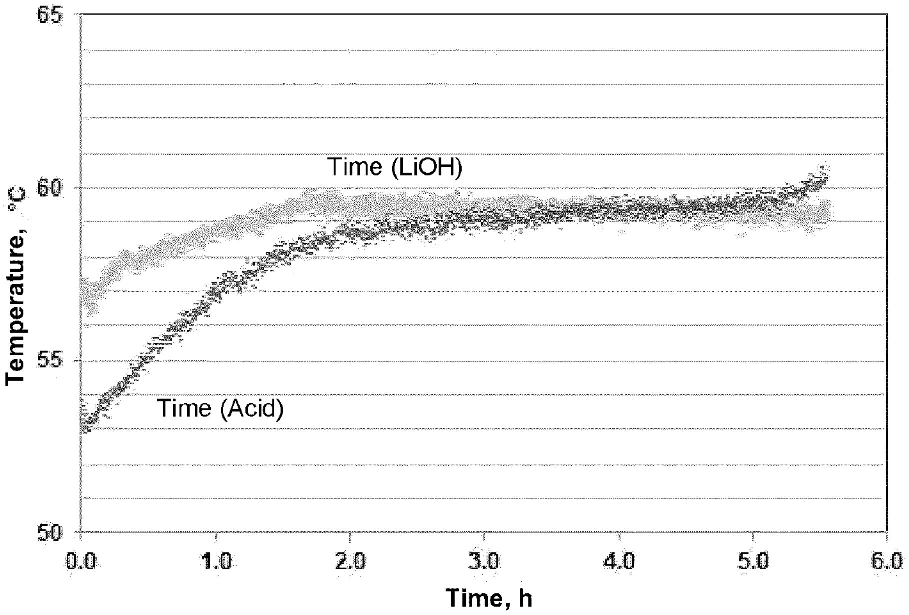

LiOH (Catholyte)
Acid (Anolyte)
Conductivity
400
350
300
250
200
150
100
50
0
0.0
1.0
2.0
3.0
4.0
5.0
6.0
Time, h

FIG. 16



65
60
55
50
Temperature, °C
Time (LiOH)
Time (Acid)
0.0
1.0
2.0
3.0
4.0
5.0
6.0
Time, h

FIG. 17

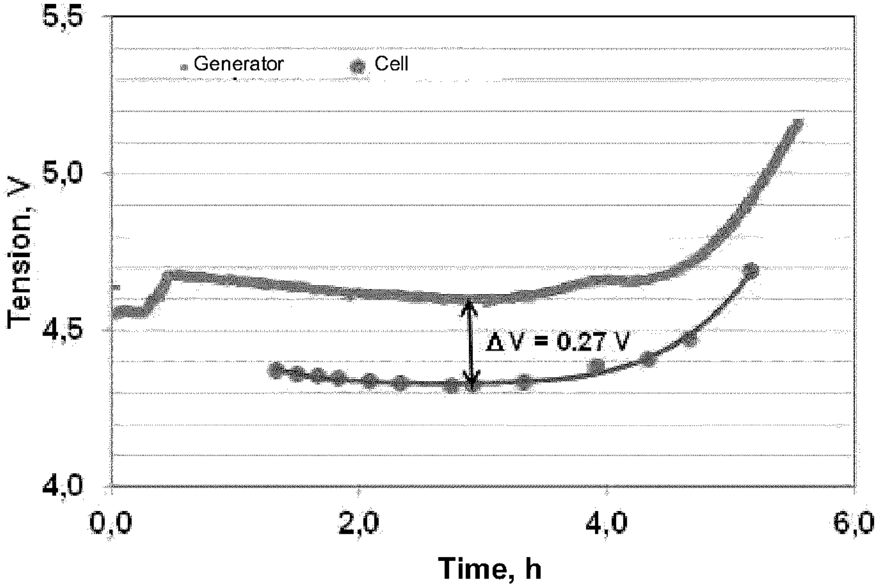


FIG. 18

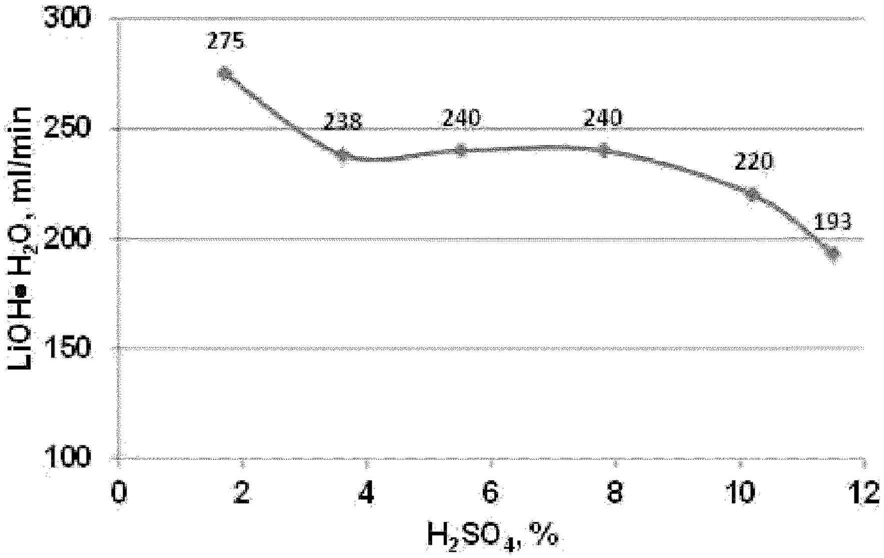

300
250
200
150
100
LiOH• H_2O, ml/min
0
2
4
6
8
10
12
H_2SO_4, %
275
238
240
240
220
193

FIG. 19

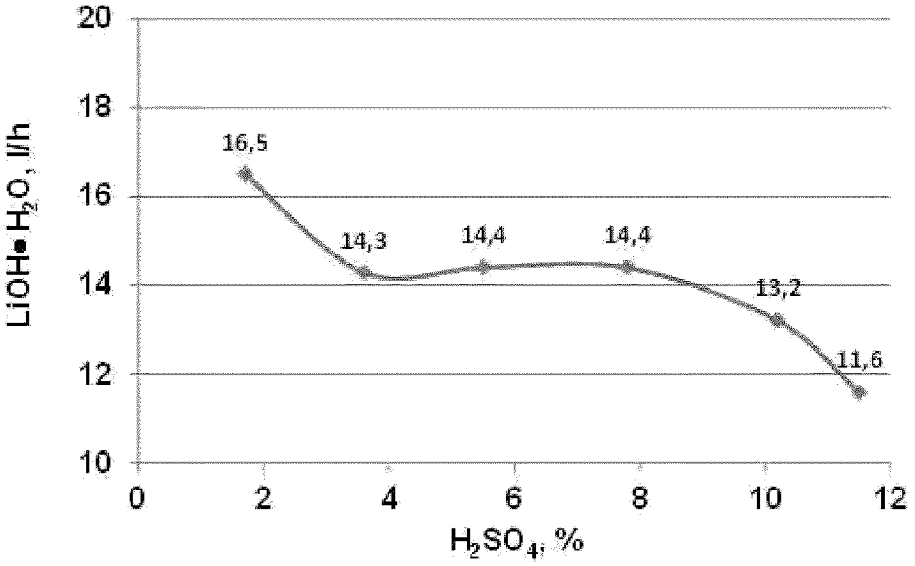

20
18
16
14
12
10
0
2
4
6
8
10
12
$LiOH\bullet H_2O$, l/h
H_2SO_4, %
16,5
14,3
14,4
14,4
13,2
11,6

FIG. 20

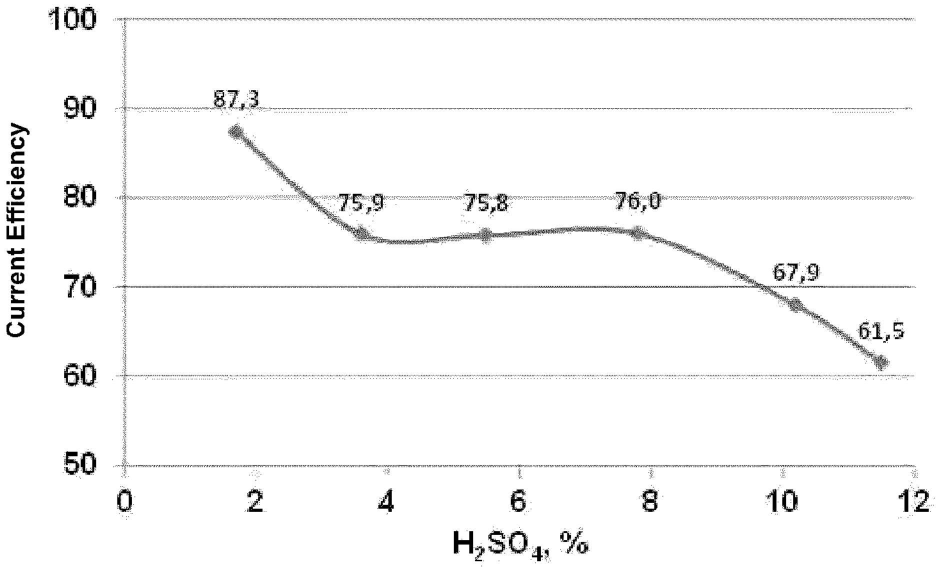

100
90
80
70
60
50
Current Efficiency
87,3
75,9
75,8
76,0
67,9
61,5
0
2
4
6
8
10
12
H_2SO_4, %

FIG. 21

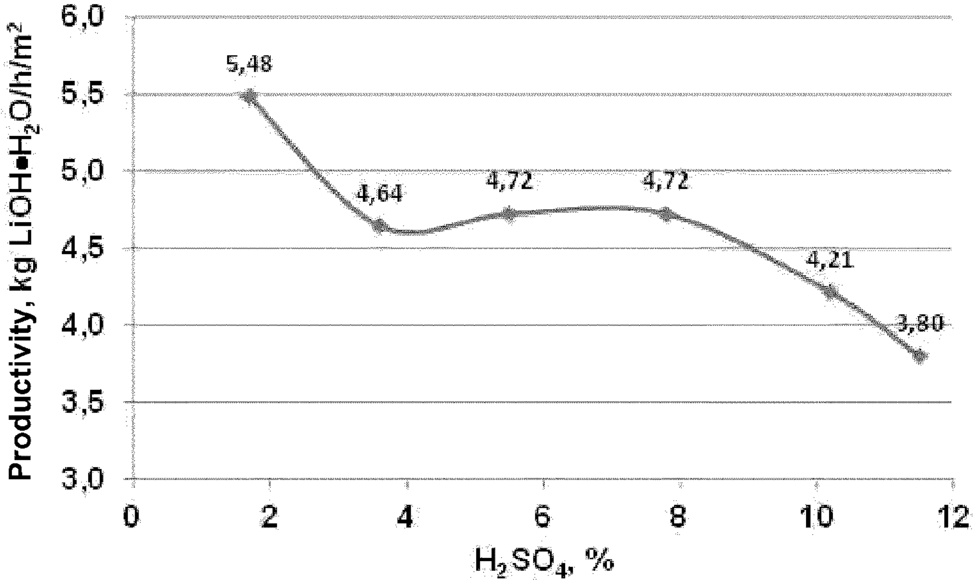

Productivity, kg LiOH•H_2O/h/m^2
6,0
5,5
5,0
4,5
4,0
3,5
3,0
0
2
4
6
8
10
12
H_2SO_4, %
5,48
4,64
4,72
4,72
4,21
3,80

FIG. 22

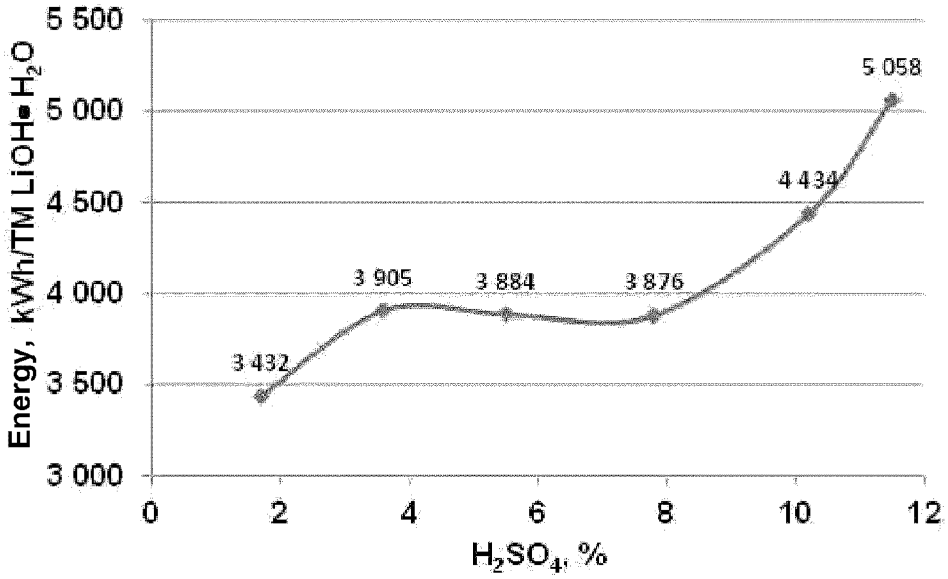

Energy, kWh/TM LiOH•H_2O
5 500
5 000
4 500
4 000
3 500
3 000
0
2
4
6
8
10
12
H_2SO_4, %
3 432
3 905
3 884
3 876
4 434
5 058

FIG. 23

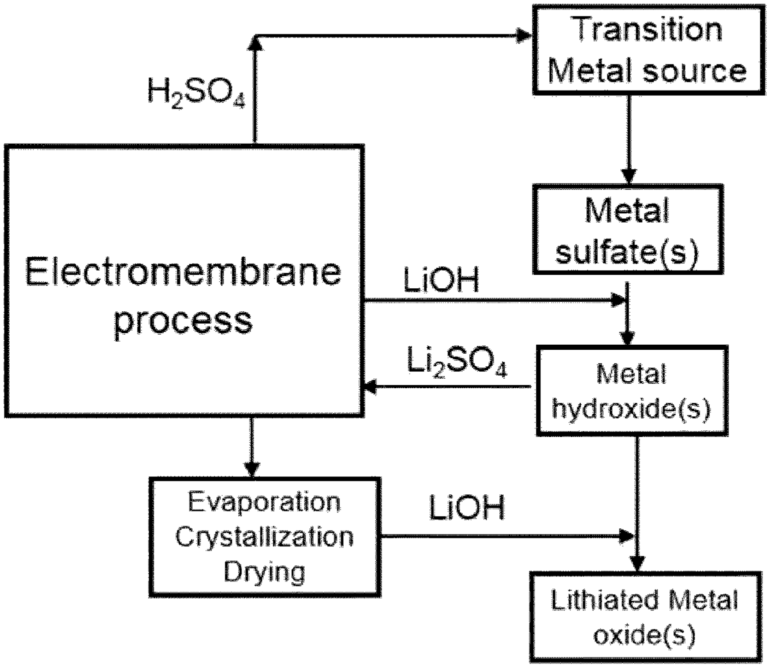

Transition Metal source
H_2SO_4
Electromembrane process
Metal sulfate(s)
LiOH
Li_2SO_4
Metal hydroxide(s)
Evaporation Crystallization Drying
LiOH
Lithiated Metal oxide(s)

FIG. 24

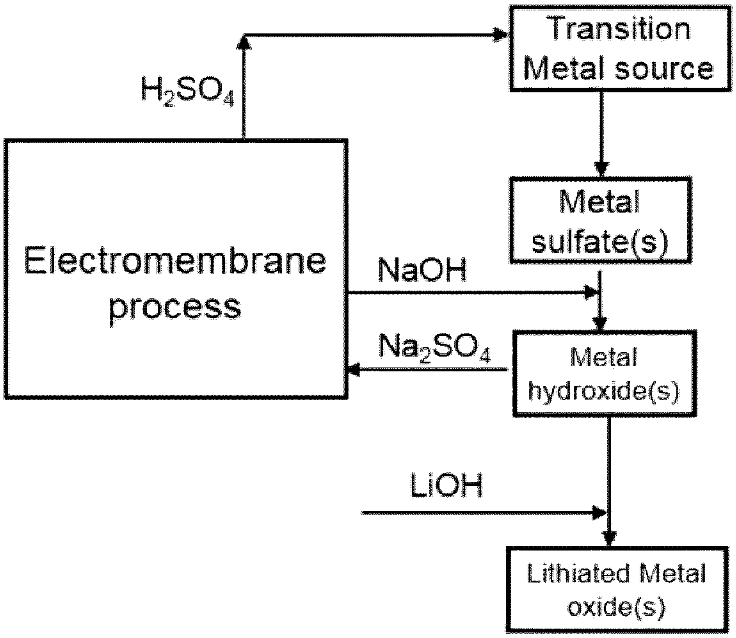

Transition Metal source
H_2SO_4
Metal sulfate(s)
Electromembrane process
NaOH
Na_2SO_4
Metal hydroxide(s)
LiOH
Lithiated Metal oxide(s)

FIG. 25

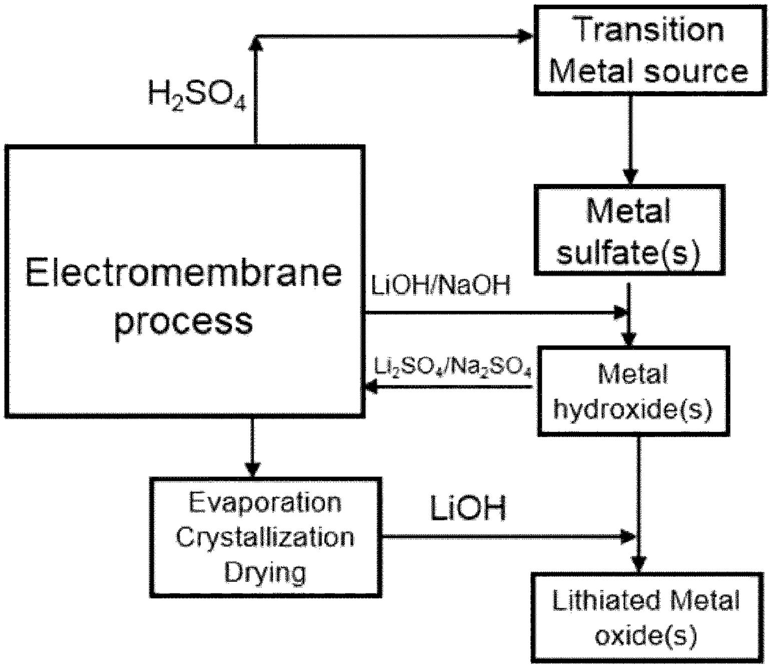

Transition Metal source
H2SO4
Electromembrane process
Metal sulfate(s)
LiOH/NaOH
Li2SO4/Na2SO4
Metal hydroxide(s)
Evaporation Crystallization Drying
LiOH
Lithiated Metal oxide(s)

FIG. 26

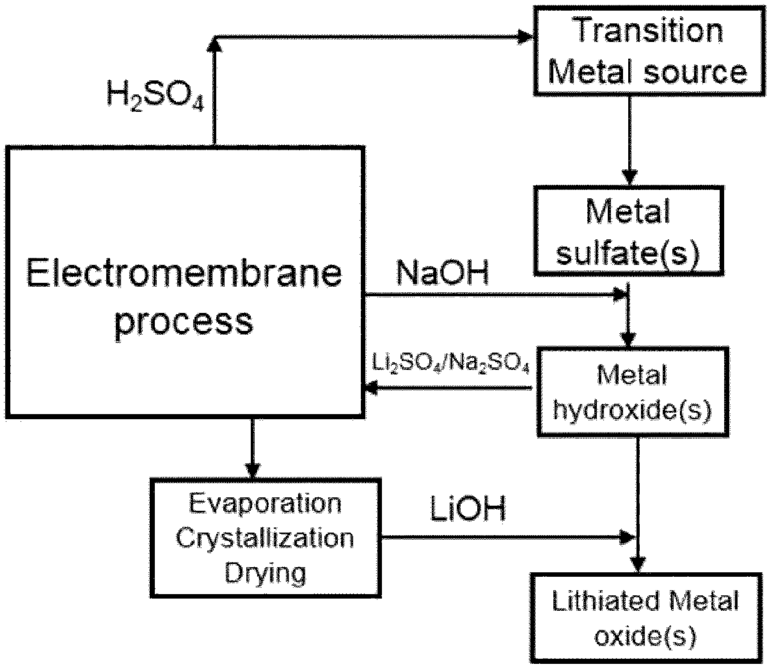

Transition Metal source
H_2SO_4
Metal sulfate(s)
Electromembrane process
NaOH
Li_2SO_4/Na_2SO_4
Metal hydroxide(s)
Evaporation Crystallization Drying
LiOH
Lithiated Metal oxide(s)

FIG. 27

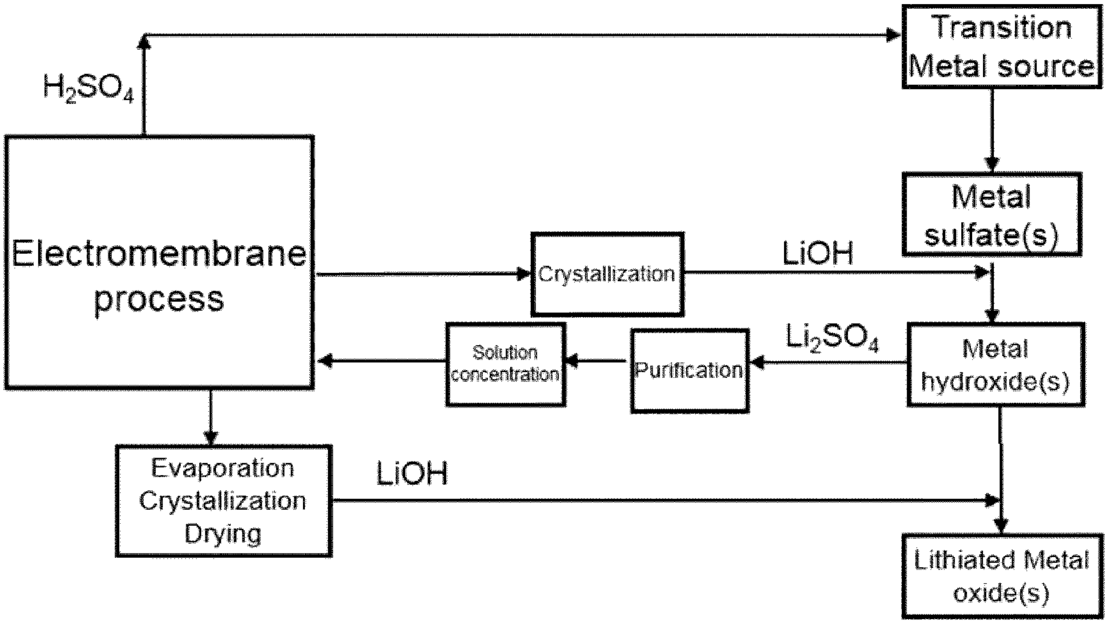

Transition Metal source
H_2SO_4
Electromembrane process
Metal sulfate(s)
Crystallization
LiOH
Li_2SO_4
Solution concentration
Purification
Metal hydroxide(s)
Evaporation Crystallization Drying
LiOH
Lithiated Metal oxide(s)

FIG. 28

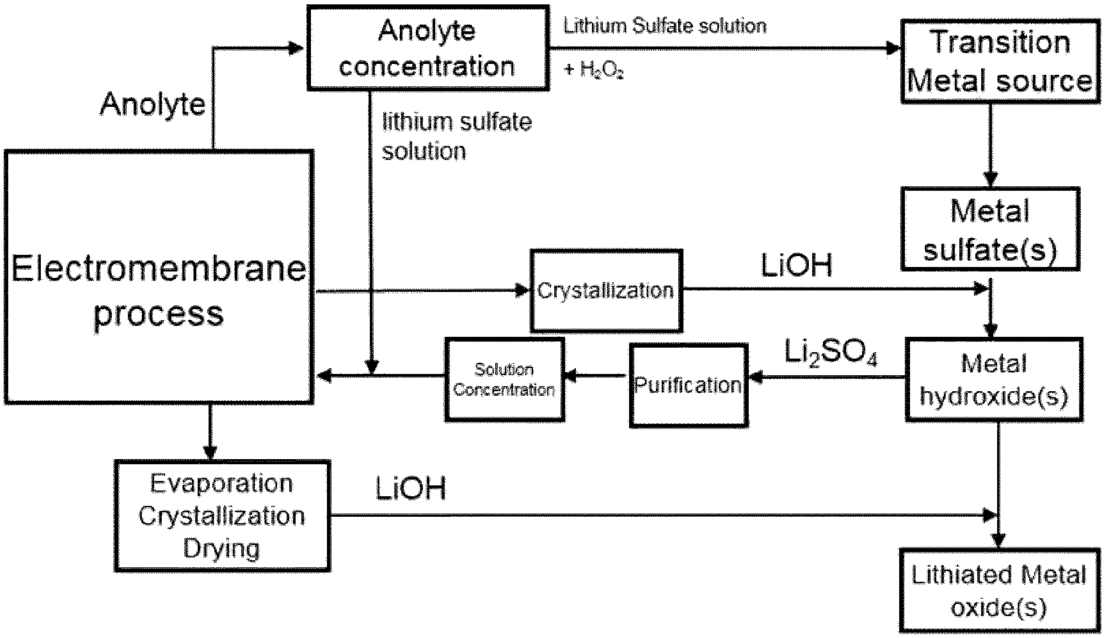

Anolyte
Anolyte concentration
Lithium Sulfate solution
+ H_2O_2
Transition Metal source
lithium sulfate solution
Metal sulfate(s)
Electromembrane process
Crystallization
LiOH
Li_2SO_4
Solution Concentration
Purification
Metal hydroxide(s)
Evaporation Crystallization Drying
LiOH
Lithiated Metal oxide(s)

FIG. 29

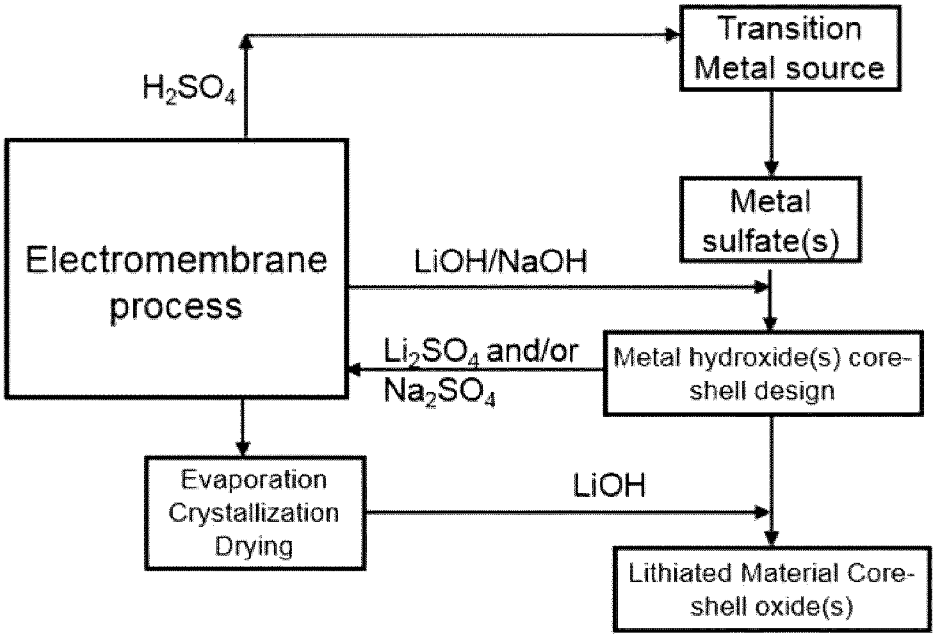

Transition Metal source
H_2SO_4
Metal sulfate(s)
Electromembrane process
LiOH/NaOH
Li_2SO_4 and/or Na_2SO_4
Metal hydroxide(s) core-shell design
Evaporation Crystallization Drying
LiOH
Lithiated Material Core-shell oxide(s)

FIG. 30

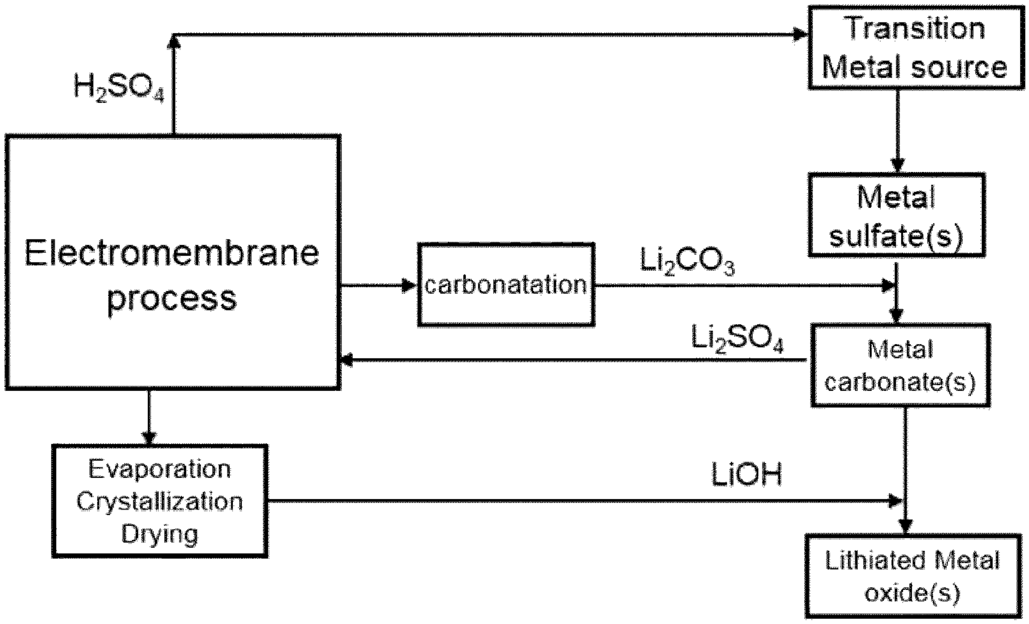

Transition Metal source
H_2SO_4
Metal sulfate(s)
Electromembrane process
carbonatation
Li_2CO_3
Li_2SO_4
Metal carbonate(s)
Evaporation Crystallization Drying
LiOH
Lithiated Metal oxide(s)

FIG. 31

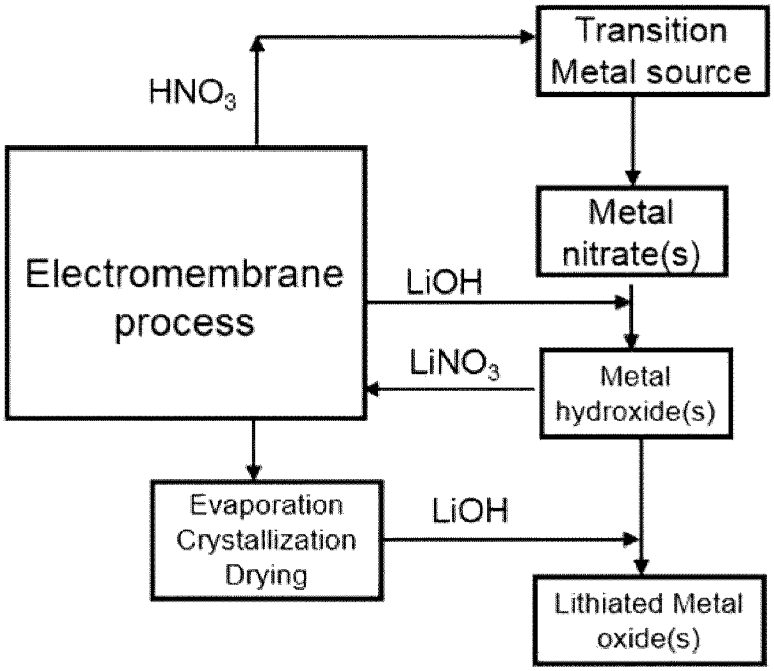

Transition Metal source
HNO3
Metal nitrate(s)
Electromembrane process
LiOH
LiNO3
Metal hydroxide(s)
Evaporation Crystallization Drying
LiOH
Lithiated Metal oxide(s)

FIG. 32

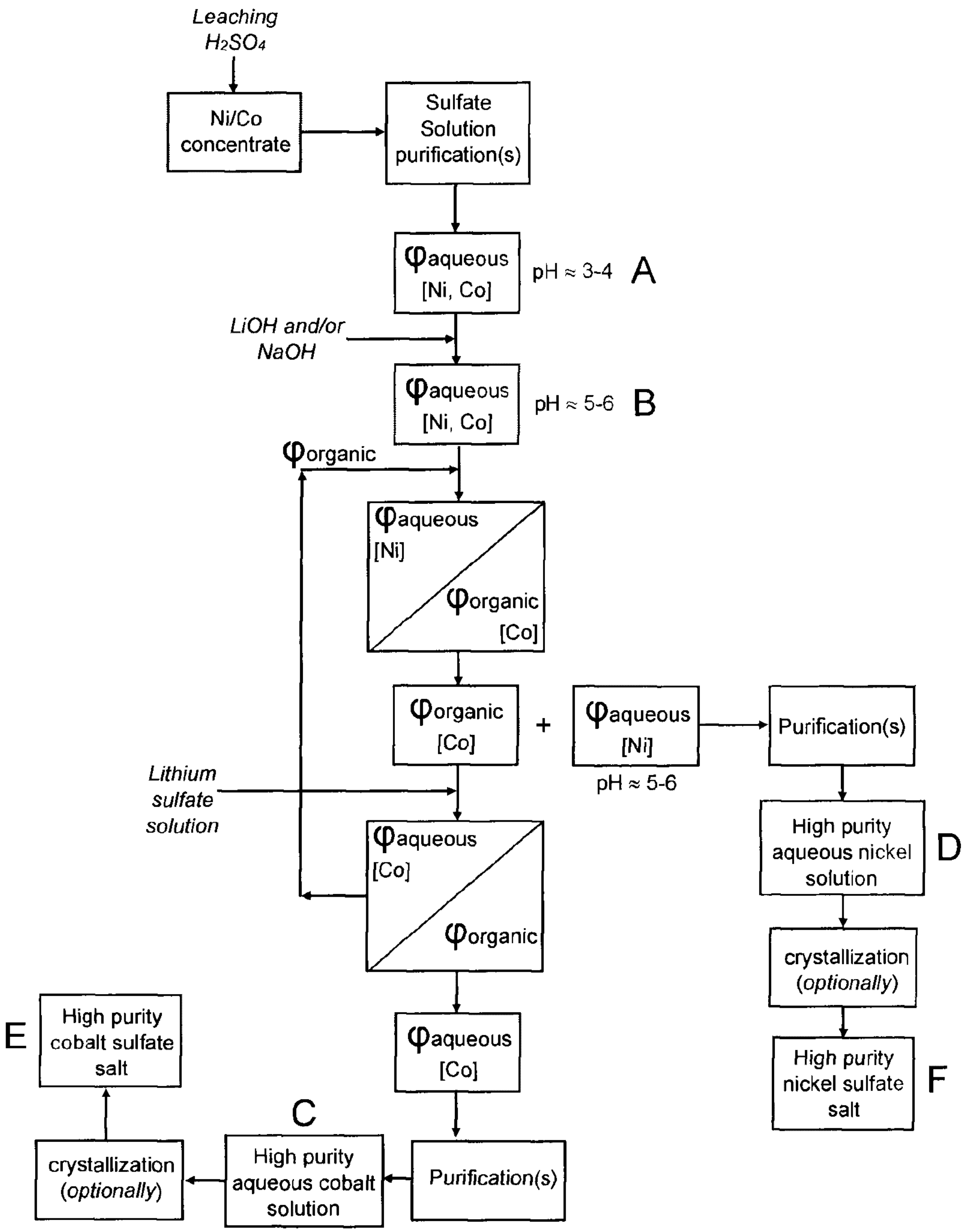

Leaching
H_2SO_4
Ni/Co concentrate
Sulfate Solution purification(s)
φaqueous [Ni, Co]
pH ≈ 3-4
A
LiOH and/or NaOH
φaqueous [Ni, Co]
pH ≈ 5-6
B
φorganic
φaqueous [Ni]
φorganic [Co]
φorganic [Co]
+
φaqueous [Ni]
pH ≈ 5-6
Purification(s)
Lithium sulfate solution
φaqueous [Co]
φorganic
High purity aqueous nickel solution
D
crystallization (optionally)
High purity nickel sulfate salt
F
φaqueous [Co]
Purification(s)
C
High purity aqueous cobalt solution
crystallization (optionally)
E
High purity cobalt sulfate salt

FIG. 33

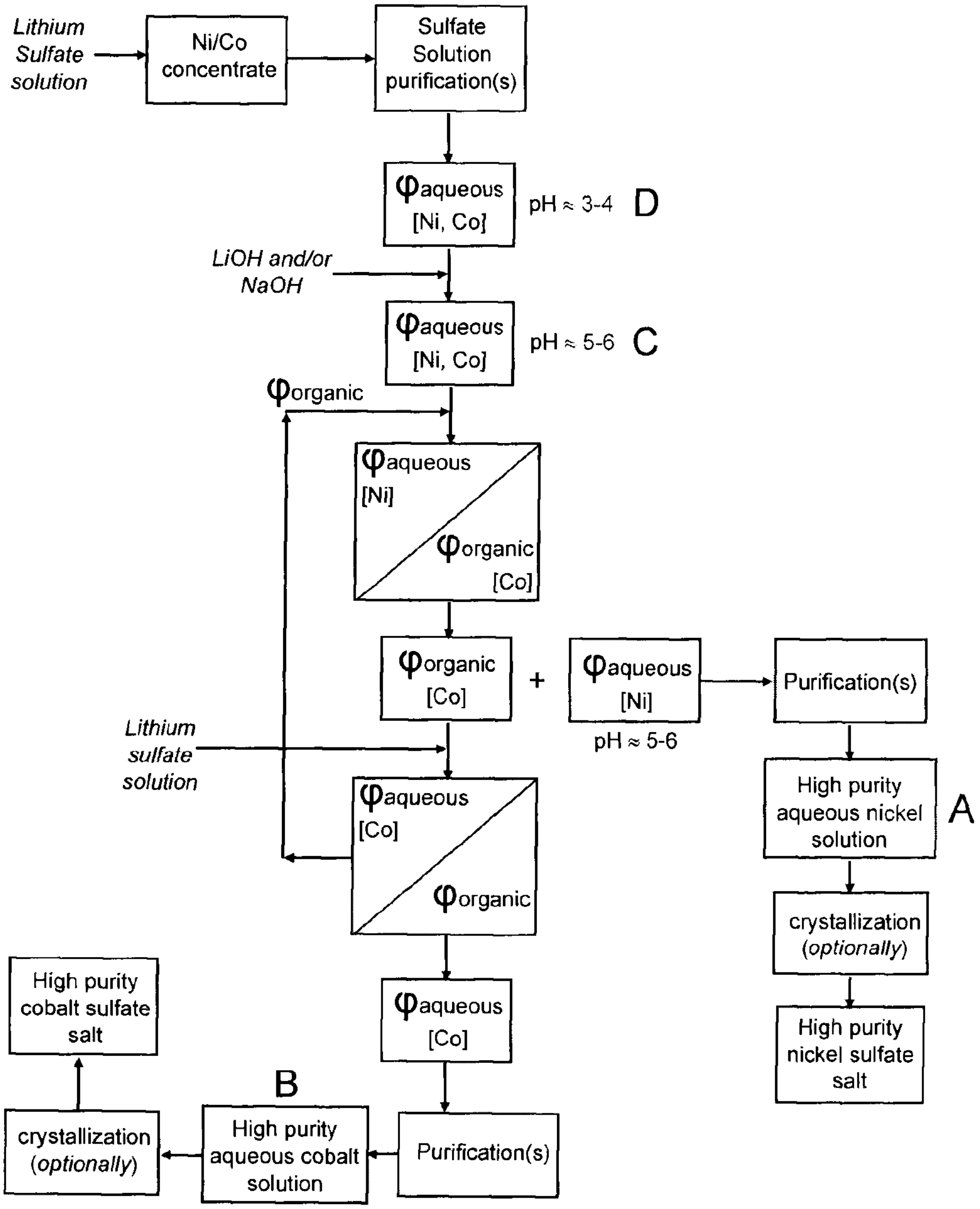

Lithium Sulfate solution
Ni/Co concentrate
Sulfate Solution purification(s)
φaqueous [Ni, Co]
pH ≈ 3-4
D
LiOH and/or NaOH
φaqueous [Ni, Co]
pH ≈ 5-6
C
φorganic
φaqueous [Ni]
φorganic [Co]
φorganic [Co]
+
φaqueous [Ni]
pH ≈ 5-6
Purification(s)
Lithium sulfate solution
φaqueous [Co]
φorganic
High purity aqueous nickel solution
A
crystallization (optionally)
High purity nickel sulfate salt
High purity cobalt sulfate salt
φaqueous [Co]
B
crystallization (optionally)
High purity aqueous cobalt solution
Purification(s)

FIG. 34

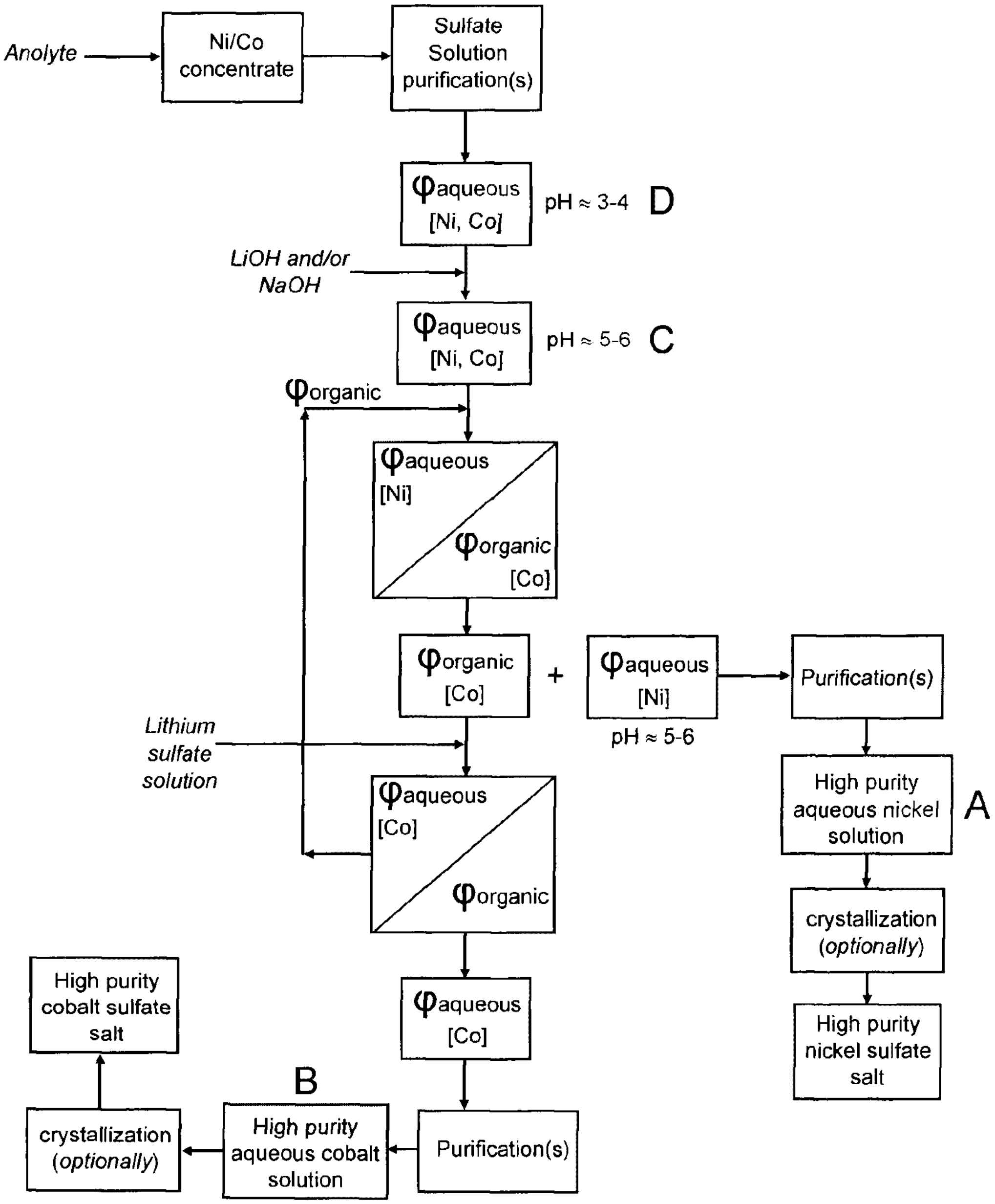
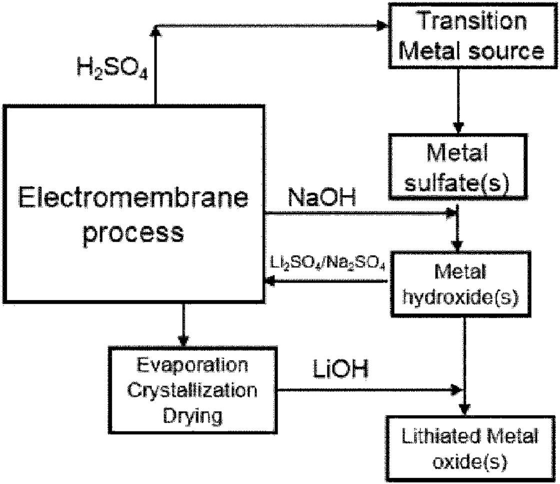

Anolyte
Ni/Co concentrate
Sulfate Solution purification(s)
φaqueous [Ni, Co]
pH ≈ 3-4
D
LiOH and/or NaOH
φaqueous [Ni, Co]
pH ≈ 5-6
C
φorganic
φaqueous [Ni]
φorganic [Co]
φorganic [Co]
+
φaqueous [Ni]
pH ≈ 5-6
Purification(s)
Lithium sulfate solution
High purity aqueous nickel solution
A
φaqueous [Co]
φorganic
crystallization (optionally)
High purity nickel sulfate salt
High purity cobalt sulfate salt
φaqueous [Co]
B
crystallization (optionally)
High purity aqueous cobalt solution
Purification(s)

FIG. 35


H$_2$SO$_4$
Transition Metal source
Metal sulfate(s)
Electromembrane process
NaOH
Li$_2$SO$_4$/Na$_2$SO$_4$
Metal hydroxide(s)
Evaporation Crystallization Drying
LiOH
Lithiated Metal oxide(s)