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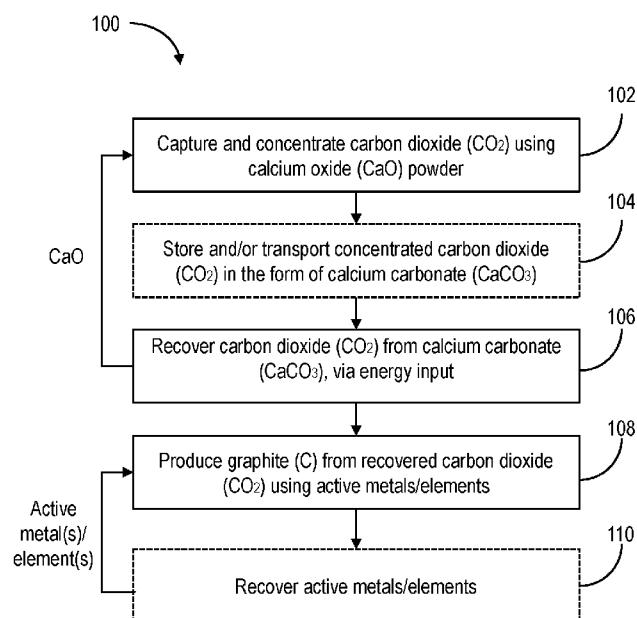


FIG. 1

(57) Abstract: Embodiments herein generally relate to a process for capture of carbon dioxide (CO₂) and conversion to graphite or graphene. In at least one embodiment, the process comprises: capturing carbon dioxide (CO₂) using calcium oxide (CaO) to produce calcium carbonate (CaCO₃); recovering the carbon dioxide (CO₂) from the calcium carbonate (CaCO₃); and reacting the carbon dioxide (CO₂) with an active metal to produce graphite (C).

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PROCESS FOR CAPTURE AND CONVERSION OF CARBON DIOXIDE

CROSS-RELATED APPLICATIONS

[0001] The present application claims the priority benefit of United States Provisional
5 Application 63/487,510, filed on February 28, 2023, the entire contents of which are incorporated herein by reference.

FIELD

[0002] The present invention generally relates to carbon capture technology, and more
10 particularly, to a process for capture of carbon dioxide (CO₂) and conversion to graphite or graphene.

BACKGROUND

[0003] Carbon capture technology has been proposed as a possible solution to mitigate the harmful effects of CO₂ emissions. Currently, many existing carbon dioxide capture or removal
15 technologies employ adsorption-storage approaches. In most cases, carbon dioxide is only temporarily captured and stored in weak and unstable systems, such as carbon dioxide captured by mining tailings, zeolites, or activated carbon, which has the possibility of carbon dioxide releasing or escaping after certain time.

SUMMARY OF VARIOUS EMBODIMENTS

[0004] In one broad aspect, there is provided a process for capture and conversion of carbon dioxide, comprising: capturing carbon dioxide (CO₂) using calcium oxide (CaO) to produce calcium carbonate (CaCO₃); recovering the carbon dioxide (CO₂) from the calcium carbonate (CaCO₃); and reacting the carbon dioxide (CO₂) with an active metal to produce graphite (C).

25 [0005] In some examples, recovering the carbon dioxide (CO₂) from the calcium carbonate (CaCO₃) is performed using calcination.

[0006] In some examples, the active metal is one of zinc (Zn), tin (Sn), manganese (Mn), Chromium (Cr), calcium (Ca), magnesium (Mg), aluminum (Al), titanium (Ti) and silicon (Si).

[0007] In some examples, an oxide of the active metal is produced as a byproduct of reacting the carbon dioxide (CO₂) with the active metal, and the process further comprises: recovery of the active metal from its oxide using a hydrometallurgy processes.

[0008] In some examples, the hydrometallurgy process comprises acid leaching followed by electrowinning.

[0009] In some examples, the recovered active metal is reused by reacting it with additional carbon dioxide (CO₂) to produce additional graphite (C).

[0010] In some examples, the calcium oxide (CaO) is generated as a byproduct of recovering the carbon dioxide (CO₂) from the calcium carbonate (CaCO₃), and the process further comprises: reusing the calcium oxide (CaO) to capture additional carbon dioxide (CO₂).

[0011] In some examples, prior to recovery of the carbon dioxide (CO₂), the process further comprises one or more of storing and transporting the captured and concentrated carbon dioxide (CO₂).

[0012] In some examples, the calcium oxide (CaO) comprises solid particles in a range of 1 nm to 100 microns.

[0013] In another broad aspect, there is provided a process for capture and conversion of carbon dioxide, comprising: capturing carbon dioxide (CO₂) using a metal oxide to produce a carbonate; recovering the carbon dioxide (CO₂) from the carbonate; and reacting the carbon dioxide (CO₂) with an active metal to produce graphite (C).

[0014] In some examples, the metal oxide is selected from the group consisting of MgO, FeO, MnO, ZnO, CuO, CaO, and mixtures thereof.

[0015] In some examples, recovering the carbon dioxide (CO₂) from the calcium carbonate is performed using calcination.

[0016] In some examples, the active metal is one of zinc (Zn), tin (Sn), manganese (Mn), Chromium (Cr), calcium (Ca), magnesium (Mg), aluminum (Al), titanium (Ti) and silicon (Si).

[0017] In some examples, an oxide of the active metal is produced as a byproduct of reacting the carbon dioxide (CO₂) with the active metal, and the process further comprises recovery of the active metal from its oxide using a hydrometallurgy processes.

[0018] In some examples, the hydrometallurgy process comprises acid leaching followed by electrowinning.

[0019] In some examples, the recovered active metal is reused by reacting it with additional carbon dioxide (CO₂) to produce additional graphite (C).

5 [0020] In some examples, the metal oxide is generated as a byproduct of recovering the carbon dioxide (CO₂) from the carbonate, and the process further comprises reusing the metal oxide to capture additional carbonate.

[0021] In some examples, prior to recovery of the carbon dioxide (CO₂), the process further comprises one or more of storing and transporting the captured and concentrated carbon dioxide (CO₂).

10 [0022] Other features and advantages of the present application will become apparent from the following detailed description taken together with the accompanying drawings. It should be understood, however, that the detailed description and the specific examples, while indicating preferred embodiments of the application, are given by way of illustration only, since various changes and modifications within the spirit and scope of the application will become apparent to those skilled
15 in the art from this detailed description.

BRIEF DESCRIPTION OF THE DRAWINGS

[0023] For a better understanding of the various embodiments described herein, and to show more clearly how these various embodiments may be carried into effect, reference will be made, by
20 way of example, to the accompanying drawings which show at least one example embodiment, and which are now described. The drawings are not intended to limit the scope of the teachings described herein.

[0024] Figure 1 is an example process for capture of carbon dioxide (CO₂) and conversion to graphite.

25 [0025] Further aspects and features of the example embodiments described herein will appear from the following description taken together with the accompanying drawings.

DETAILED DESCRIPTION OF THE EMBODIMENTS**I. DEFINITIONS**

[0026] Any term or expression not expressly defined herein shall have its commonly accepted definition understood by a person skilled in the art. As used herein, the following terms have the following meanings.

[0027] "Active metals" refer to a class of metals, known in the art, that react strongly and quickly with other elements due to the electrons in their structure, and their ease of sharing the electrons with other elements. Examples of active metals include zinc (Zn), tin (Sn), manganese (Mn), Chromium (Cr), calcium (Ca), magnesium (Mg), aluminum (Al), titanium (Ti) and silicon (Si).

[0028] "Carbon capture" refers to the process of trapping and storing carbon (e.g., carbon dioxide in the atmosphere, often produced by burning of fossil fuels or other chemicals).

[0029] "Graphite" is an opaque, non-metallic carbon polymorph, represented by the element symbol (C).

[0030] "Graphene" is a one atomic layer of carbon atoms tightly bound in a hexagonal honeycomb lattice. It is an allotrope of carbon and is considered a form of graphite.

[0031] "Hydrometallurgy process" is a technique within the field of extractive metallurgy, involving the obtaining of metals from their ores. Hydrometallurgy involve the use of aqueous solutions for the recovery of metals from ores, concentrates, and recycled or residual material.

II. GENERAL OVERVIEW

[0032] Embodiments described herein generally relate to a process for capture of carbon dioxide (CO₂) and the production of graphite or graphene.

[0033] The disclosed process involves capturing carbon dioxide (CO₂) using calcium oxide (CaO) powder, which is believed to provide an efficient and stable method for CO₂ capture. The

process then subsequently involves conversion of the captured and concentrated carbon dioxide (CO₂) to graphite/graphene, using an active metal (e.g., Zn, Sn, Mn, Cr, Ca, Mg, Al, Ti, or Si).

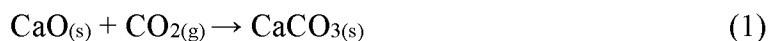
[0034] The process combines the objectives of CO₂ reduction with the production of graphite. Graphite is considered to be an important, highly valued, but fast depleting mineral, which has broad applications in modern industries such as electrode materials in lithium batteries and electrometallurgy. In some embodiments, graphene is produced, which is also a valuable product.

[0035] It is believed that the disclosed process mitigates drawbacks associated with conventional carbon capture technology. More broadly, the disclosed process is regarded as relatively safe, technologically feasible and economically viable.

III. EXAMPLE PROCESS

[0036] Figure 1 shows an example process (100) for capture of carbon dioxide (CO₂) and conversion to graphite (or graphene).

[0037] As shown, at act (102), CO₂ is captured by reacting the CO₂ with calcium oxide (CaO) powder. This results in the production of solid calcium carbonate (CaCO₃), according to the reaction in Equation (1):



[0038] More generally, it has been appreciated that CaO is an efficient CO₂ capturer and concentrator. This is because the CaCO₃ forming reaction, expressed in Equation (1), can occur quickly and thoroughly. In some examples, higher specific areas or smaller particle sizes, of CaO powder (e.g., nano powder), may be more efficient for CO₂ capture, e.g., because of the increased surface area of the smaller particles. In at least one example, the grain sizes of CaO used in Equation (1) are in a range of 1 nm to 100 microns, which can be built/fixed together into certain shapes (e.g., bee hives). It has been appreciated that the smaller the CaO powder, the more efficient CO₂ capture. In some examples, the CO₂ capturing process in Equation (1) operates at the temperature and pressure of the flue gas containing CO₂. The CaO structure for CO₂ capture can be replaced after a certain

elapsed time to maintain enough remaining active CaO, which may be determined by the characteristics of the flue gas, e.g., flow rate and CO₂ content.

[0039] The disclosed embodiments are not limited to the environment, or context, in which the CO₂ can be captured, in accordance with the reaction in Equation (1). For example, CO₂ may be captured from the atmosphere (e.g., air). For instance, floating objects (e.g., balloons) may be loaded with CaO powder, and used to deduct CO₂ concentration. In air, the concentration of CO₂ is typically low (e.g., less than 1%), as air is typically comprised of 78% N₂ and 21% O₂. Accordingly, CO₂ capturing and concentration in the form of calcium carbonate is necessary for efficient CO₂ fixing and eventual conversion to carbon (graphite).

[0040] CO₂ can also be captured from the exhaust gas of vehicles (e.g., vehicles using hydrocarbon fuels). For instance, CaO may be incorporated into the vehicle exhaust pipes to capture CO₂. In still other examples, the CO₂ can be captured from industrial flue gases, such as those from power plants fueled by coal or oils. In these examples, the CaO powder can be used via the blow reaction. For instance, CaO packed columns can be installed to remove CO₂ from the industrial flue gases.

[0041] At act (104), in some examples, the captured and concentrated carbon dioxide (CO₂) – in the form of calcium carbonate (CaCO₃) – is stored away until a time where it is required for producing graphite or graphene. In other cases, it may also be transported to a designated site, or location, where the CO₂ can be recovered with a view to producing graphite or graphene. For instance, the CaCO₃ can be transported using any type of vehicle, or otherwise any suitable transport or conveyance mechanism.

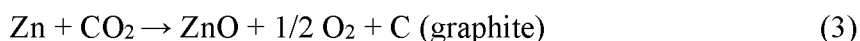
[0042] To that end, the storage and/or transport of the captured and concentrated carbon dioxide (CO₂) is possible because calcium carbonate (CaCO₃) provides a strong and stable capture medium that concentrates CO₂ for extended time periods.

[0043] At act (106), the captured and concentrated CO₂, in the form of CaCO₃, is recovered or released via input energy. This can be preformed using calcination (heating), in accordance with the reaction in Equation (2):



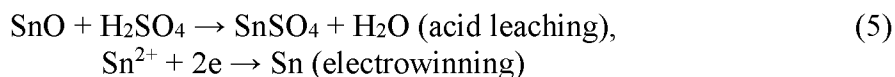
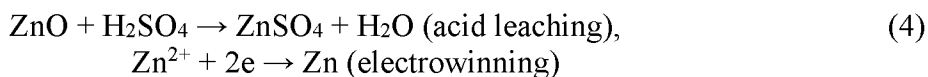
[0044] In some examples, the CaO, produced by the calcination process, is returned to the first step, to be re-used to capture additional CO₂. This is expressed by the return line, in process (100). Accordingly, CaO can be recycled in the process (100) in order to save CO₂ capturing and minimize solid waste discharge. In at least one example, the calcination temperature in Equation (2) is set in a range of between 800°C to 900°C under ambient pressures or certain vacuum environments, to remove and collect CO₂ produced out of the calcination system.

[0045] At act (108), the released CO₂, which is almost pure gas, can be converted to graphite (C) by reducing it with an active metal. Equation (3) shows an example reaction using zinc (Zn) as the active metal.



[0046] It will be understood that Equation (3) is a thermodynamically favorable reaction. From an Ellingham diagram, thermodynamically, both CO and CO₂ can be reduced to graphite (C) by more active metals/elements (e.g., Zn, Mn, Cr, Ca, Mg, Al, Ti, Si, and Mn), when the oxidation line of the metal or element is below the formation lines of CO and CO₂, under certain temperatures. Active metal tin (Sn) can also reduce CO and CO₂ to C (graphite) based on its half cell electrode potential. From a carbon phase diagram, the thermodynamically stable phase for carbon under relatively low pressure is graphite.

[0047] At act (110), in some examples, the active metals, used in Equation (3), can be recovered and recycled from their oxides using hydrometallurgy processes (e.g., acid leaching followed by electrowinning), as known in the art. Equations (4) and (5) show examples of this recovery process for zinc (Zn) and tin (Sn).



[0048] Accordingly, the recovered active metal can be re-used in Equation (3). This is expressed by the return line, in process (100). As metals can be recycled economically from the

metal oxides, the presented process for fixing graphite (C) from CO₂, using active metals, is considered to be an economically viable and technologically feasible process.

IV. ALTERNATIVE EXAMPLES

5 [0049] While FIG. 1 (act (102)) and Equation (1) exemplify a case where carbon dioxide (CO₂) is captured using calcium oxide (CaO) powder, in other examples, it is appreciated that carbon dioxide (CO₂) can also be captured using any other metal oxide to produce a corresponding carbonate. In at least one example, the metal oxide is selected from a group consisting of MgO, FeO, MnO, ZnO, CuO, CaO, or any mixture thereof. In these examples, at act (106) (FIG. 1) and Equation (2), heating can
10 also be used in a similar manner to recover the carbon dioxide (CO₂) from the carbonate. At act (108), and Equation (3), carbon dioxide (CO₂) can be reacted with an active metal to produce graphite (C).

V. EXPERIMENTAL SET-UP

[0050] To demonstrate the efficacy of the process (100), an experimental set-up was
15 arranged, whereby CO₂ gas flow was blown through a ceramic crucible filled with an active metal to obtain C (graphite, characterizing with XRD). Example parameters and conditions for converting CO₂ to graphite/graphene using various active metals are listed in Table 1, below.

Table 1 – Carbon Fixing by Blowing CO₂ Gas through an Active Metal

Metal used for converting CO₂ to graphite	Temperature, °C	CO₂ gas flow, liter/minute	Conversion time, minutes	Carbon conversion efficiency CE
Zn	500	100	5	10.2%
	600	60	20	30.6%
Sn	280	100	20	7.6%
	400	100	15	27.5%
Al	780	100	20	18.6%
	900	80	15	33.9%

Ti	1000	100	120	1.6%
	1780	80	15	26.7%
Si	900	120	120	1.9%
	1780	80	120	7.8%

[0051] The carbon conversion efficiency (CE) can be calculated according to Equation (6).

$$CE = m_{\text{graphite}}/m_{\text{carbon-in-carbon dioxide}} \quad (6)$$

wherein m_{graphite} is the mass of graphite obtained after acid leaching and drying; $m_{\text{carbon-in-carbon dioxide}}$ is the mass of carbon in CO₂ gas flow within certain conversion time. Carbon mass in CO₂ gas flow may
5 determined from CO₂ mass using the ideal gas equation under stable volumic gas flow, in fixed conversion time (e.g., 1 to 120 minutes).

VI. INTERPRETATION

[0052] Various systems or methods have been described to provide an example of an
10 embodiment of the claimed subject matter. No embodiment described limits any claimed subject matter and any claimed subject matter may cover methods or systems that differ from those described below. The claimed subject matter is not limited to systems or methods having all of the features of any one system or method described below or to features common to multiple or all of the apparatuses or methods described below. It is possible that a system or method described is not an embodiment
15 that is recited in any claimed subject matter. Any subject matter disclosed in a system or method described that is not claimed in this document may be the subject matter of another protective instrument, for example, a continuing patent application, and the applicants, inventors or owners do not intend to abandon, disclaim or dedicate to the public any such subject matter by its disclosure in this document.

20 [0053] Furthermore, it will be appreciated that for simplicity and clarity of illustration, where considered appropriate, reference numerals may be repeated among the figures to indicate corresponding or analogous elements. In addition, numerous specific details are set forth in order to

provide a thorough understanding of the embodiments described herein. However, it will be understood by those of ordinary skill in the art that the embodiments described herein may be practiced without these specific details. In other instances, well-known methods, procedures and components have not been described in detail so as not to obscure the embodiments described herein. Also, the description is not to be considered as limiting the scope of the embodiments described herein.

[0054] It should also be noted that the terms “coupled” or “coupling” as used herein can have several different meanings depending in the context in which these terms are used. For example, the terms coupled or coupling may be used to indicate that an element or device can electrically, optically, or wirelessly send data to another element or device as well as receive data from another element or device. As used herein, two or more components are said to be “coupled”, or “connected” where the parts are joined or operate together either directly or indirectly (i.e., through one or more intermediate components), so long as a link occurs. As used herein and in the claims, two or more parts are said to be “directly coupled”, or “directly connected”, where the parts are joined or operate together without intervening intermediate components.

[0055] It should be noted that terms of degree such as "substantially", "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 may also be construed as including a deviation of the modified term if this deviation would not negate the meaning of the term it modifies.

[0056] Furthermore, any recitation of numerical ranges by endpoints herein includes all numbers and fractions subsumed within that range (e.g. 1 to 5 includes 1, 1.5, 2, 2.75, 3, 3.90, 4, and 5). It is also to be understood that all numbers and fractions thereof are presumed to be modified by the term "about" which means a variation of up to a certain amount of the number to which reference is being made if the end result is not significantly changed.

[0057] The present invention has been described here by way of example only, while numerous specific details are set forth herein in order to provide a thorough understanding of the exemplary embodiments described herein. However, it will be understood by those of ordinary skill in the art that these embodiments may, in some cases, be practiced without these specific details. In other instances, well-known methods, procedures and components have not been described in detail so as not to obscure the description of the embodiments. Various modification and variations may be

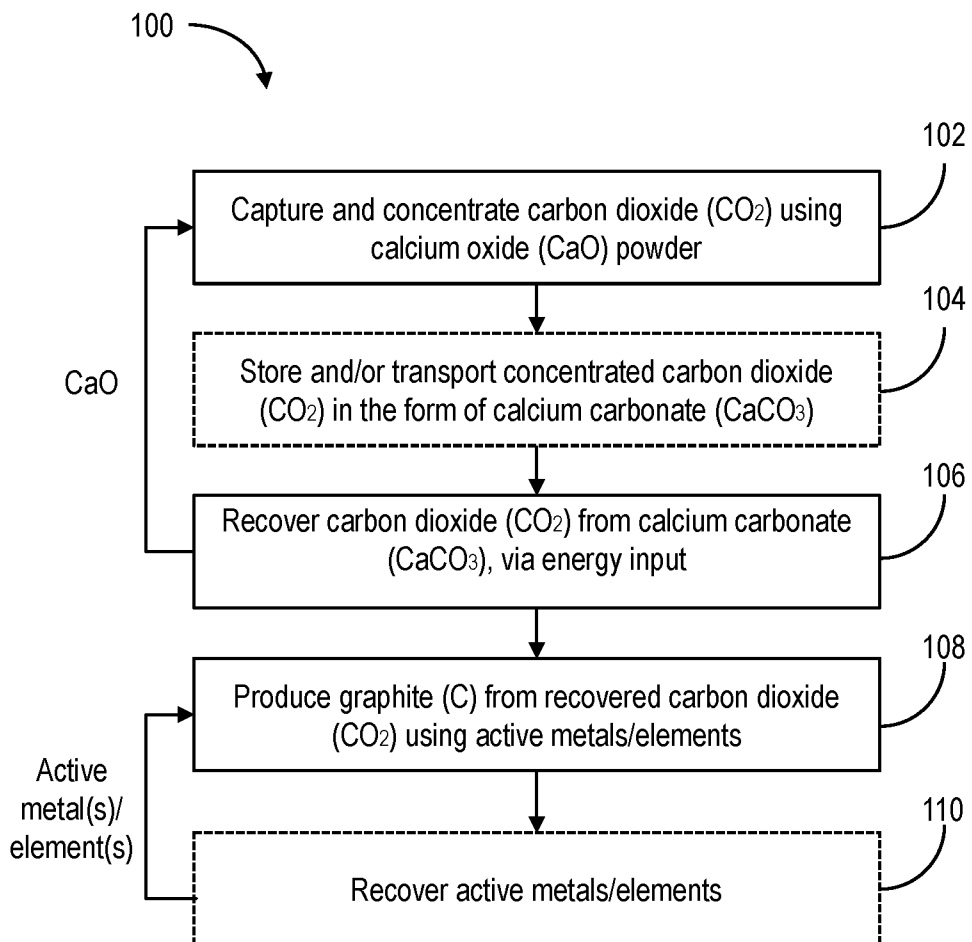
made to these exemplary embodiments without departing from the spirit and scope of the invention, which is limited only by the appended claims.

CLAIMS:

1. A process for capture and conversion of carbon dioxide, comprising:
capturing carbon dioxide (CO₂) using calcium oxide (CaO) to produce calcium carbonate
5 (CaCO₃);
recovering the carbon dioxide (CO₂) from the calcium carbonate (CaCO₃); and
reacting the carbon dioxide (CO₂) with an active metal to produce graphite (C).
2. The process of claim 1, wherein recovering the carbon dioxide (CO₂) from the calcium carbonate (CaCO₃) is performed using calcination.
- 10 3. The process of any one of claims 1 or 2, wherein the active metal is selected from the group consisting of zinc (Zn), tin (Sn), manganese (Mn), Chromium (Cr), calcium (Ca), magnesium (Mg), aluminum (Al), titanium (Ti) and silicon (Si).
4. The process of any one of claims 1 to 3, wherein an oxide of the active metal is produced as a byproduct of reacting the carbon dioxide (CO₂) with the active metal, and the process further
15 comprises recovery of the active metal from its oxide using a hydrometallurgy processes.
5. The process of claim 4, wherein the hydrometallurgy process comprises acid leaching followed by electrowinning.
6. The process of claims 4 or 5, wherein the recovered active metal is reused by reacting it with additional carbon dioxide (CO₂) to produce additional graphite (C).
- 20 7. The process of any one of claims 1 to 6, wherein calcium oxide (CaO) is generated as a byproduct of recovering the carbon dioxide (CO₂) from the calcium carbonate (CaCO₃), and the process further comprises reusing the calcium oxide (CaO) to capture additional carbon dioxide (CO₂).
8. The process of any one of claims 1 to 7, wherein prior to recovery of the carbon dioxide (CO₂), the process further comprises one or more of storing and transporting the captured and concentrated
25 carbon dioxide (CO₂).

9. The process of any one of claims 1 to 8, wherein the calcium oxide (CaO) comprises solid particles in a range of 1 nm to 100 microns.
10. A process for capture and conversion of carbon dioxide, comprising:
5 capturing carbon dioxide (CO₂) using a metal oxide to produce a carbonate;
recovering the carbon dioxide (CO₂) from the carbonate; and
reacting the carbon dioxide (CO₂) with an active metal to produce graphite (C).
11. The process of claim 10, wherein the metal oxide is selected from the group consisting of
10 MgO, FeO, MnO, ZnO, CuO, CaO, and mixtures thereof.
12. The process of claims 10 or 11, wherein recovering the carbon dioxide (CO₂) from the calcium carbonate is performed using calcination.
13. The process of any one of claims 10 to 12, wherein the active metal is selected from the group consisting of zinc (Zn), tin (Sn), manganese (Mn), Chromium (Cr), calcium (Ca), magnesium (Mg),
15 aluminum (Al), titanium (Ti) and silicon (Si).
14. The process of any one of claims 10 to 13, wherein an oxide of the active metal is produced as a byproduct of reacting the carbon dioxide (CO₂) with the active metal, and the process further comprises recovery of the active metal from its oxide using a hydrometallurgy processes.
15. The process of claim 14, wherein the hydrometallurgy process comprises acid leaching
20 followed by electrowinning.
16. The process of claims 14 or 15, wherein the recovered active metal is reused by reacting it with additional carbon dioxide (CO₂) to produce additional graphite (C).
17. The process of any one of claims 10 to 16, wherein metal oxide is generated as a byproduct of recovering the carbon dioxide (CO₂) from the carbonate, and the process further comprises reusing the
25 metal oxide to capture additional carbonate.

18. The process of any one of claims 10 to 17, wherein prior to recovery of the carbon dioxide (CO₂), the process further comprises one or more of storing and transporting the captured and concentrated carbon dioxide (CO₂).

**FIG. 1**

INTERNATIONAL SEARCH REPORT

International application No.

PCT/CA2024/050248

A. CLASSIFICATION OF SUBJECT MATTER

IPC: **B01D 53/62** (2006.01), **B01D 53/14** (2006.01)

CPC: , B01D 53/14 (2020.01), B01D 53/62 (2020.01)

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC: **B01D 53/62** (2006.01), **B01D 53/14** (2006.01) CPC: B01D 53/14 (2020.01), B01D 53/62 (2020.01)

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

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

World Wide Web, Questel-Orbit

Keywords:

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	Hawthorne, C. et al., "Simulation of the carbonate looping power cycle", Energy Procedia, volume 1, pages 1387-1394, 2009 * the whole document *	1-3, 7-13, 17-18
Y	Li, X. et al., "Direct synthesis of graphene by blowing CO ₂ bubble in Mg melt for the seawater/oil pollution, Journal of Alloys and Compounds, volume 921, pages 1-10, 15 November 2022 (15-11-2022) * the whole document *	1-3, 7-13, 17-18

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

* "A" "D" "E" "L" "O" "P"	Special categories of cited documents: document defining the general state of the art which is not considered to be of particular relevance document cited by the applicant in the international application earlier application or patent but published on or after the international filing date document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) document referring to an oral disclosure, use, exhibition or other means document published prior to the international filing date but later than the priority date claimed	"T" "X" "Y" "&"	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art document member of the same patent family
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Date of the actual completion of the international search
17 April 2024 (17-04-2024)Date of mailing of the international search report
17 April 2024 (17-04-2024)Name and mailing address of the ISA/CA
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