**Supporting Information**

# Multiscale process intensification of waste valorization reactions


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## INTRODUCTION (complementary to main text)

The four research streams developed to study complex slurry-phase reactions, namely “brine carbonation”, “mineral carbonation and enhanced weathering”, “process intensification and integration” and “characterization techniques”, are interrelated as follows.

*Brine carbonation* work was undertaken in view of searching for faster routes to $CO_2$ mineralization. Given that in brine carbonation the alkaline earth metals are already dissolved and available for precipitating as carbonates, the leaching step of mineral carbonation and enhanced weathering reactions is eliminated, although additives are required to counter the acidification of the reaction medium. As such, there was extensive knowledge transfer from prior work done on mineral carbonation by Dr. Santos in 2010-2016 (which is not covered in this ACS Account) that was critical to guide the recent work on brine carbonation.

Under the stream of *mineral carbonation and enhanced weathering*, the work conducted in 2017-2023 looked at advancing prior work, with a greater focus on matching the right feedstock and the right additive to the right process, in view of increasing reaction efficiency (i.e., targeting valorization and net $CO_2$ sequestration above simply kinetics and extent of reaction). Notably, knowledge gained from brine carbonation work has served to guide enhanced weathering work in terms of investigating pedogenic carbonate formation and the carbon drawdown by mine tailing ponds in recent research of Dr. Santos.

*Process intensification and integration* (PI&I) are key strategies employed in conceptualizing and designing carbonation and weathering processes that combine waste valorization. This is where principles of engineering aid in scaling up and maximizing the net carbon negativity of fundamental geochemical reactions. As such, works that incorporated PI&I are strongly interlinked to more fundamental studies covered in the aforementioned streams, but they have their thematics that seek to enable the industrial uptake of carbonation and weathering processes.

All of the aforementioned streams include research that uses advanced instruments to inform of their efficacy and the properties of their feedstocks, products, and by-products. The *characterization techniques* stream highlights work that shows which techniques are underutilized and how they can inform mechanistic aspects of carbonation and weathering reactions that are less evident without the purposeful combination of characterizations and the interpretation of their results.

**RESEARCH STREAMS (complementary to main text)**

The three limitations of complex slurry-phase reactions can be defined as follows: (i) "reaction rate" pertains to how fast mineral carbonation or waste conversion reactions go; (ii) "conversion" pertains to how much of a material's reaction potential is realized, basically as a measure of the extent of reaction; and (iii) "efficiency" pertains to minimizing energy or chemicals expenditures and the generation of new wastes or emissions. Based on these definitions, several papers included within the 18 highlighted articles[1-18] (which include the 4 key references[4,8,12,18] and their derivative works) cover these research aspects in several ways. Of the 18 highlighted papers, 11 of them match these research objectives, as summarized below:

- Ali et al. (2023)[2]: focus on *conversion* in terms of assessing the purity of the sodium bicarbonate target product and the avoidance of the formation of undesirable secondary phases, and on *efficiency* in terms of evaluating various additives that can replace the conventional use of volatile ammonia.
- Chai et al. (2021a)[3]: focus on *reaction rate* by studying the weathering and carbonation of two distinctively reactive silicates under a range of reaction intensities.
- Chai et al. (2021b)[4]: focus on *conversion* by monitoring the X-ray diffraction intensities of silicate and carbonate phases in-situ during carbonation reactions, and also consider *efficiency* in terms of exposing silicate minerals to subcritical and supercritical carbonation absent of a liquid phase to assess the effectiveness of these alternate carbonation routes.
- Chalouati et al. (2023)[5]: the use of a ball mill reactor was meant to evaluate the effect of continuous comminution during mineral carbonation on the *reaction rate* and *conversion*, and the testing of various additives aimed to assess the most chemically-*efficient* route.
- Fantucci et al. (2021)[8]: catalytic wet oxidation of fruit processing residue combined with mine tailings was shown to benefit the upgrading of these wastes in terms of improved *reaction rate*, *conversion*, and *efficiency* when compared to conventional composting and anaerobic digestion processes.

- Georgakopoulos et al. (2017)[11]: the generation of two valuable products from metallurgical slag, combined with $CO_2$, without producing additional wastes or emissions exemplifies a waste valorization process that achieves high *conversion* and *efficiency*.
- Hicks et al. (2022)[13]: this perspective introduces the concept of synthesizing fast weathering silicates for enhanced weathering application in cold and dry climates, as a means to overcome *reaction rate* and *conversion* limitations experienced in these climatic settings, and moreover proposes integrating the production of these nutrient-doped silicates with cement production and geological $CO_2$ sequestration and partly replacing carbon-positive conventional fertilizers as a means of improving the *efficiency* of agricultural production.
- Ounoughene et al. (2018)[14]: the study addresses the *efficiency* challenges of direct air capture, which is a technology known to have large energy demand, and accomplishes this by evaluating a novel solvochemical route for direct air capture.
- Zhang et al. (2019)[18]: in this first of a series of three studies on brine carbonation, the use of nickel nanoparticles is proposed as means to increase the *reaction rate* and the *conversion* of $CO_2$ mineralization in a tubular reactor of short residence time.
- Zhang et al. (2020b)[17]: in this second study of the brine carbonation series, MEA replaces NaOH to improve the process *efficiency* by facilitating the recovery of the additives from the spent brine.
- Zhang et al. (2021)[16]: in this third iteration of brine carbonation, the $CO_2$ mineralization process is re-imagined by introducing dechlorination rather than pH buffering as a means to induce carbonate precipitation, and to this end regenerable hydrotalcite is proposed and tested, along with a comprehensive investigation of the process *efficiency*, the *reaction rate* and the reaction *conversion*.

## SCIENTOMETRIC ANALYSIS (complementary to main text)

As of August 15th 2023, the 18 journal articles highlighted in this ACS Account (as related to the topic of “Multiscale process intensification of waste valorization reactions”)[1-18] that were published between 2017 and 2023 had been cited according to Web of Science (WoS) a total of 169 times (157 without self-citations) by 157 articles (149 without self-citation). Important to note here is that the definition of self-citation here means the exclusion of any of the 18 highlighted articles of Dr. Santos from the citation count, which means that some of Dr. Santos’s group’s other articles are not part of this selection of 18 (in this period, Dr. Santos published a total of 65 journal articles) are not counted as self-citation. **Fig. S1** presents the yearly publication and citation counts of 17 of the 18 highlighted articles (1 is not included as it was still in-press at the time of this analysis, so not yet indexed in WoS).

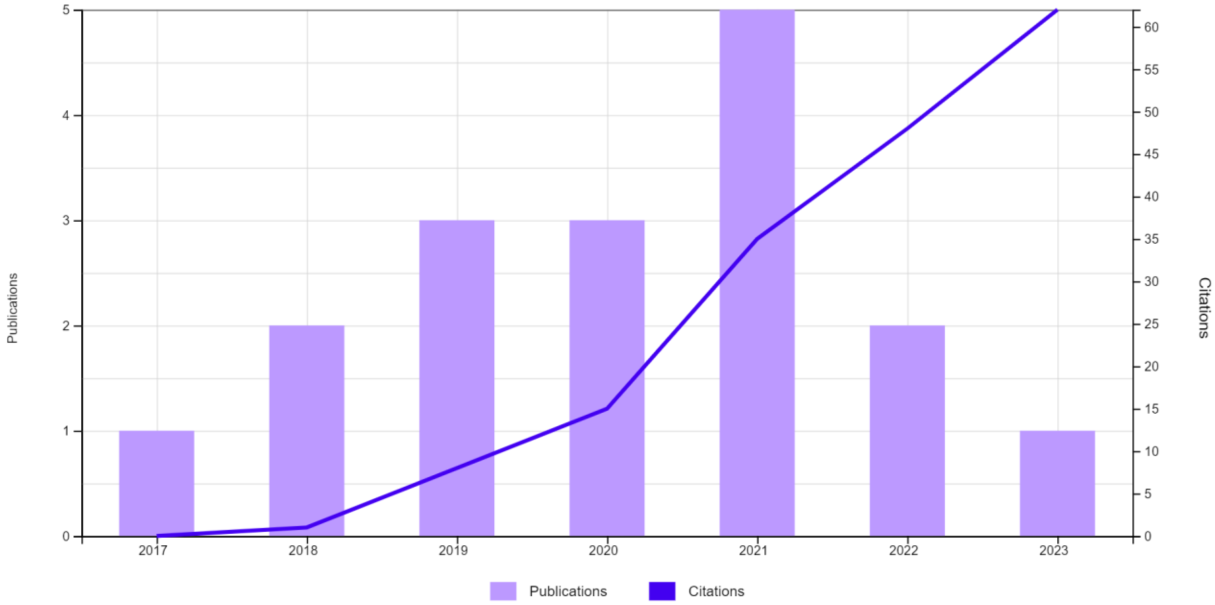


**Fig. S1.** Publication and total citation count of 17 of the 18 highlighted articles of Dr. Santos. Data source: WoS (August 15th 2023).

**Citation Usage**

WoS recently added a new set of scientometric data to its records called the Citing Items Breakdown. It intends to classify each citation of a cited article based on the reason for its use by a citing article. To this end, WoS defined five reasons why an article may be cited by another article:

- *Background*: When the cited article's research serves to orient the study of the citing article.
- *Basis*: When a citing article uses data sets, methods, concepts, or ideas from the cited article, either directly or indirectly.
- *Support*: Where the cited article mentions that its results or methods are similar to those of the citing article, including attempts to replicate the prior work.
- *Differ*: Where the cited article mentions that its results, methods, or sample sizes differ from those of the citing article in such a manner that results in differing outcomes.
- *Discuss*: A cited article that mentions the citing article in its detailed discussions.

(notation): "cited" means the article that was cited in the citing article; "citing" means the article that is citing the cited article.

**Fig. S2** presents the aggregated data from the 17 WoS-indexed articles highlighted in this ACS Account. It should be noted that this dataset is partial. WoS counted 55 citations for this analysis, out of the 168 total, likely because its automated article crawler was only able to conclusively discern a select number of cases. Still, it is insightful to inspect this data, wherein the classification of *Background* is the most common, and there are no records for *Differ*. It is very common to cite an article that has been influential in setting the stage of a research article in its introduction section, which WoS counts as Background. *Basis* is the next most common occurrence, showing that close to a third of the citing articles used the cited items to more specifically guide or shape the new research, while over a quarter of the citations refer to *Discuss*, where the citing articles used the previous research to explain trends or mechanism found in their own research. The low number of cases for Support (only 1 out of 55 occurrences) may signify a difficulty in an automated system to discern this type of citation use (perhaps it is limited to looking for citations given within tables or captions). What is also insightful is how this type of new scientometric data is an example of what may be to come as Web of Science and other scholarly databases incorporate artificial intelligence into their services, as recently reported by Van Noorden (2023)[19].

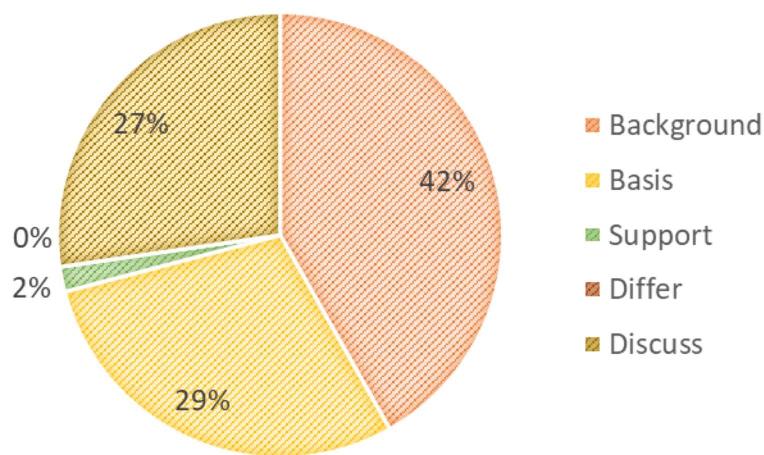


**Fig. S2.** Citing Items Breakdown data of 17 of the 18 highlighted articles of Dr. Santos. Data source: WoS (August 11th 2023).

We also conducted our own "manual" scientometric analysis of citing items usage by selecting the most relevant citing articles (excluding review papers) that cited any of the 18 highlighted cited articles and inspecting them to infer the reasons for the citations (**Table S1**).

**Table S1.** The most relevant citing articles that cited the cited articles.

| Citing article | Paper type | Focus | Cited article | Citation usage |
|---|---|---|---|---|
| Williamson et al. (2023)[20] | Article | Carbon mineralization | Dudhaiya & Santos (2018)[7] | Introduction |
| Bortali et al. (2023)[21] | Article | Dredged sediment | Ali et al. (2022)[1] | Introduction |
| Y. Wang et al. (2022)[22] | Article | Carbon mineralization | Zhang et al. (2021)[16] | Introduction |
| Laajimi et al. (2022)[23] | Article | Ultrasound | Haque, Santos, & Chiang (2019)[12] | Introduction |
| Ruhaimi et al. (2022)[24] | Article | Porous carbonaceous | Zhang, Chai et al. (2020a)[15] | Introduction |
| Polidoro et al. (2022)[25] | Article | Carbon mineralization | Fernandez Rivas et al. (2020)[9] | Introduction |
| Behloul et al. (2022)[26] | Article | Carbon mineralization | Fernandez Rivas et al. (2020)[9] | Introduction |
| W. Li et al. (2022)[27] | Article | Carbon mineralization | Zhang, Chai et al. (2020a)[15] | Introduction |
| Cuevas et al. (2022)[28] | Article | XRD Quantitative Analysis | Ali et al. (2022)[1] | Introduction |
| Chen et al. (2022)[29] | Article | Carbon mineralization | Chai, Miller, et al. (2021)[4] | Introduction |
| Hong et al. (2022)[30] | Article | Carbon mineralization | Zhang, Chai et al. (2020a)[15] | Introduction |
| Sim et al. (2022)[31] | Article | Carbon mineralization | Zhang, Chai et al. (2020a)[15] | Introduction |
| Zheng et al. (2022)[32] | Article | Mineral carbonation | Zhang, Chai et al. (2020a)[15] | Introduction |
| J. Li, Jacobs, et al. (2022)[33] | Article | Mineral carbonation | Zhang, Chai et al. (2020a)[15] | Introduction |
| J. Wang, Watanabe et al. (2022)[34] | Article | Carbon mineralization | Zhang, Chai et al. (2020a)[15] | Introduction |
| Behloul et al. (2021)[35] | Article | Heat transfer | Fernandez Rivas et al. (2020)[9] | Introduction |

| | | | | |
|---|---|---|---|---|
| Commenge (2021)[36] | Article | Miniaturization | Fernandez Rivas et al. (2020)[9] | Introduction |
| Baciocchi & Costa (2021)[37] | Article | Mineral carbonation | Santos et al. (2012); Zhang et al. (2021)[16] | Introduction |
| Galina et al. (2023)[38] | Article | Mineral carbonation | Zhang, Chai et al. (2020a)[15] | Introduction |
| Shen et al. (2023)[39] | Article | Airborne-particle abrasion | Ali et al. (2022)[1] | Methodology |
| Kelly et al. (2023)[40] | Article | Mineralogy and Muribaculaceae | Ali et al. (2022)[1] | Methodology |
| Khalidy et al. (2021)[41] | Article | Carbon Accumulation | Dudhaiya et al. (2019)[6] | Methodology |
| Bullock et al. (2023)[42] | Article | Mineral carbonation | Dudhaiya et al. (2019)[6] | Results and discussion |
| Zhang & Moment (2023)[43] | Article | Carbon Mineralization | Zhang et al. (2019)[18] | Results and discussion |
| Micheluz et al. (2022)[44] | Article | Biophysical Manipulation | Ali et al. (2022)[1] | Results and discussion |
| J. Li, Wang, et al. (2022)[45] | Article | Mineral carbonation | Zhang, Chai et al. (2020a)[15] | Results and discussion |
| Fei et al. (2022)[46] | Article | Mineral carbonation | Zhang, Chai et al. (2020a)[15] | Results and discussion |
| Bullock et al. (2022)[47] | Article | Mine tailings | Dudhaiya et al. (2019)[6] | Results and discussion |
| Zhu et al. (2022)[48] | Article | Carbon mineralization | Zhang et al. (2021)[16] | Results and discussion |

In the introduction section of the citing articles, approximately 60% of them specifically addressed the topic of carbon mineralization and mineral carbonization. Notably, a significant portion of these papers referenced Zhang, Chai et al. (2020a)[15], which by being a review paper about scaling up these technologies is a good fit for being used as a supporting background for new research.

In the methodology section of citing articles, there was also a consistent focus on carbon mineralization and mineral carbonization among the papers that cited one of the 18 highlighted articles' experimental methodology. Shen et al. (2023)[39] and Kelly et al. (2023)[40] utilized X-ray diffraction (XRD) for characterizing crystalline structures, phase identification, transformation, and semi-quantitative mineral identifications, and as such cited the current "hot paper" (according to WoS) Ali et al. (2022)[1]. Khalidy et al. (2021)[41] is an example of a paper from Dr. Santos' group that is about enhanced rock weathering (ERW), and as such not part of the selection of 18 highlighted articles in this ACS Account, but that utilized several of the reaction and characterization methods used in many of these articles, thus citing several of them as shown in **Table S1**.

In the results section of citing articles, similar areas of focus on carbon mineralization and mineral carbonization were observed, indicating their prominence among the papers. Carbon mineralization and mineral carbonization consistently emerged as the primary areas of interest for the papers. Zhang & Moment (2023)[43] experimented to prepare various polymorphs of calcium carbonates by controlling operating conditions such as reaction temperature, base dosing method, aging time, ionic content, and supersaturation control; their findings were consistent with the results reported in Zhang et al. (2019)[18]. Micheluz et al. (2022)[44] compared their X-ray powder diffraction analysis with that of Ali et al. (2022)[1]. Zhu et al. (2022)[48] referred to the explanation provided by Zhang et al. (2021)[16] regarding the utilization of alkaline industrial waste materials, including hydrotalcite supergroup minerals from mine wastes, through anion exchange and partial dissolution. Fei et al. (2022)[46] compared the $CO_2$ removal efficiencies of different ash samples over time and related their findings to the slow kinetics of carbonation reactions and the challenges associated with the utilization of carbonated products as presented by Zhang, Chai et al. (2020a)[15].

### Citing Topics and Sources

Word clouds are compilations of words frequently found in source texts, and thus a word cloud generated from keywords of journal articles can be used to summarize the common topics of research of a select group of articles. The word cloud generated from the keywords extracted from all 148 papers that cited the 18 highlighted papers is shown in **Fig. S3**. The largely synonymous terms “mineral carbonation” and “$CO_2$ mineralization” were the two most common keywords. Other similar terms such as “carbon dioxide removal”, “$CO_2$ sequestration” and “carbon/$CO_2$ capture” appear often. More specific terms that appear relatively often include: “drug delivery”, “soil carbon”, “powder metallurgy”, “circular economy” and “process intensification”. Most of these words are well aligned with the focus of this ACS Account. The one exception is “drug delivery”, which appears frequently in the keywords due to citations to the XRD review paper Ali et al. (2022)[1] that is being widely cited by all science disciplines that use XRD analysis.

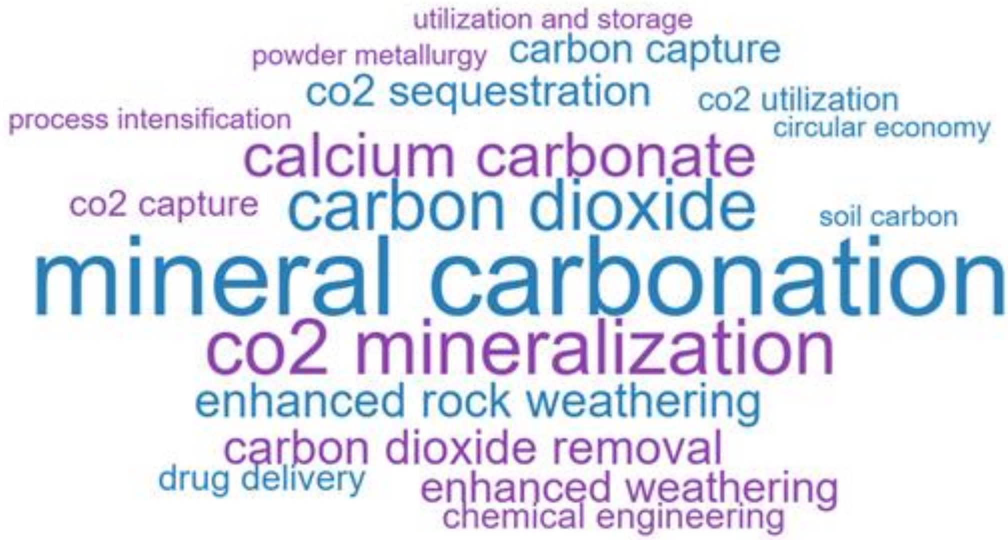


**Fig. S3.** Word cloud of keywords from the 149 citing papers. Generated using WordCloud+.

Table S2 and Fig. S4 present additional classifications of the citing articles' topics in terms of areas of research and titles of the journals, respectively. Table S2 shows an even split between applied research areas (including Engineering, Materials Science, Energy/Fuels, Construction, Metallurgy, and Mining) and fundamental research areas (including Chemistry, Environmental Sciences, Geochemistry, and Education), speaking to the multidisciplinary audience of the highlighted papers and the topics of mineral carbonation, waste valorization, and process intensification. This also explains the large number of unique journals where the 149 citing articles appear in. Within the top 11, the most frequent journals only published between 3 and 6 of these articles, led by the aptly titled Journal of $CO_2$ Utilization. Notably, when it comes to publishers, not only the top 11 list but the entire list is led by journals from Elsevier (65) and MDPI (33), with the other major publishers American Chemical Society (9), the Royal Society for Chemistry (8), Springer Nature (7), Frontiers (5) and Wiley (5) coming far behind. There appears to be a "citation-by-association" phenomenon driving this termed publisher self-citation (Zhou, 2021)[49], as 14 of the 18 highlighted cited articles were published in Elsevier (9) and MDPI (5) journals.

**Table S2.** Ranking of areas of research (classified by WoS) of the 149 citing articles. Data source: WoS (August 15th 2023)

| **Research areas** | **Number of publications** |
|---|---|
| Engineering | 58 |
| Chemistry | 45 |
| Materials Science | 33 |
| Science Technology & Other Topics | 23 |
| Environmental Sciences & Ecology | 21 |
| Energy & Fuels | 15 |
| Physics | 12 |
| Construction Building Technology; Geochemistry & Geophysics | 6 (each) |
| Education & Educational Research; Metallurgy & Metallurgical Engineering; Mining & Mineral Processing | 5 (each) |

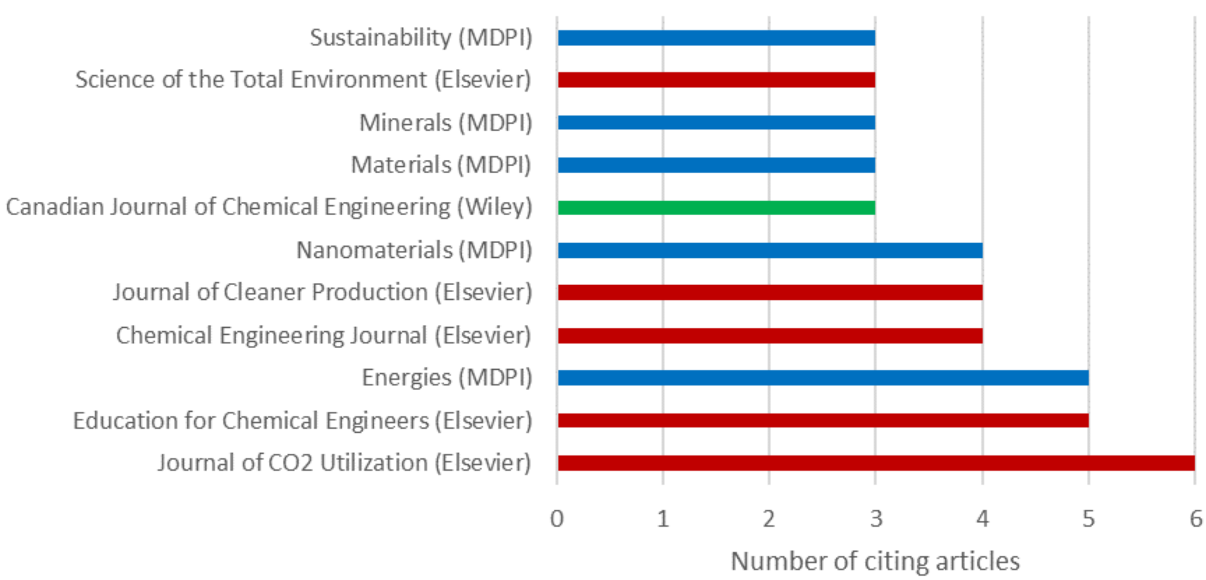


**Fig. S4.** Titles of the top 11 journals that published the 149 citing articles. Publishers are color-coded. Data source: WoS (August 15th 2023).

## Citing Country Analysis

**Fig. S5** illustrates the countries affiliated with the citing articles. Unsurprisingly the list is dominated by countries with high publication output such as China, the USA, large European countries, and other scientifically productive Asian countries. Australia and Canada appear high on the list being countries with large efforts for mineral carbonation, mine tailing management, and critical metal recovery. The main surprise in Fig. S5 could be Saudi Arabia. This is explained by 6 of the 7 papers having cited the XRD review paper of Ali et al. (2022)[1], aforementioned of being multi-disciplinary but also to be noted is that the first-author obtained their Master's degree in that country and hence that can attract an intra-country network of readers. The seventh citation is from a multi-country review paper on direct air capture materials (Wang, Fu et al., 2022)[50] that cited the solvochemical ambient carbonation paper of Ounoughene et al. (2018)[14].


**Fig. S5.** Top 15 countries of affiliation of the 149 citing articles. Data source: WoS (August 15th 2023). Note: multiple authors from the same country affiliated with the same paper count as a single country affiliation.

To highlight the multi-author nature of the citing papers, the diagram in **Fig. S6** showcases a network representation of 34 countries that have published at least one citing article that has multi-country authors. In the network, the size of a nodal circle corresponds to the number of citing articles published by that country that meet this multi-author criterion. Additionally, the thickness of the lines connecting countries indicates the level of cooperation between them. The centrality value of a country reflects its influence in the network. The nodal circle sizes more or less follow the order of the data in Fig. S5, though in a different order (in parentheses is the number of lines connecting to each country node): Canada (6), China (9), UK (7), USA (8), and Germany (6) having the top 5 nodes both in terms of circle size and number of lines. Notably, even large countries like China and USA often collaborate with authors from other countries in papers related to mineral carbonation and waste valorization, showing the value of collaborations from different points of view and expertise, especially given that mineral resources, environmental regulations, and climate change mitigation efforts vary from country to country (and continent to continent). Surprisingly Australia only shows collaborations with China in this subset despite ranking 5th in **Fig. S5**. On the other hand, Spain has 4 lines in **Fig. S6**, showing large multi-country collaboration, yet ranks 11th-tied in **Fig. S5**.

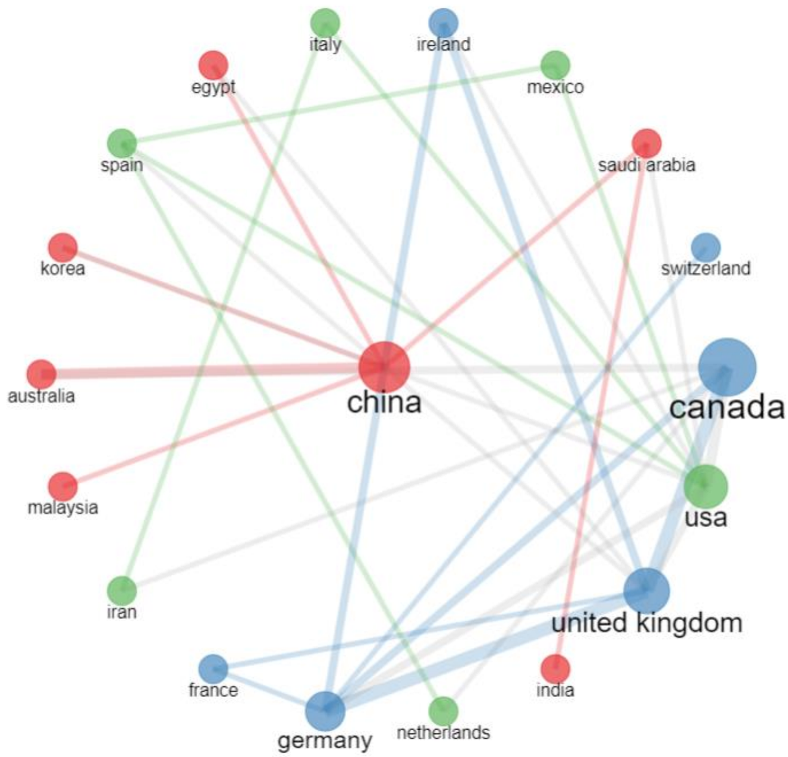


**Fig. S6.** The collaboration network of the countries with multi-author citing articles (including self-citations). Data source: WoS (August 6th 2023).

**Citing Institution Analysis**

The level of academic support and collaboration between Dr. Santos's affiliation (University of Guelph) and other institutions can be assessed by examining the institutional affiliations of multi-author citing articles, including self-citations (i.e., citations of any of the 17 WoS-indexed highlighted articles to each other). **Fig. S7** illustrates this collaboration network, with the University of Guelph at the center of the hub and having 7 lines, highlighting the large extent of inter-institutional collaboration in Dr. Santos' research. The institutions in this article set that have more than 7 lines are the University of Newcastle (9 lines), the University of Lorraine (8 lines), and the University of Twente (8 lines).

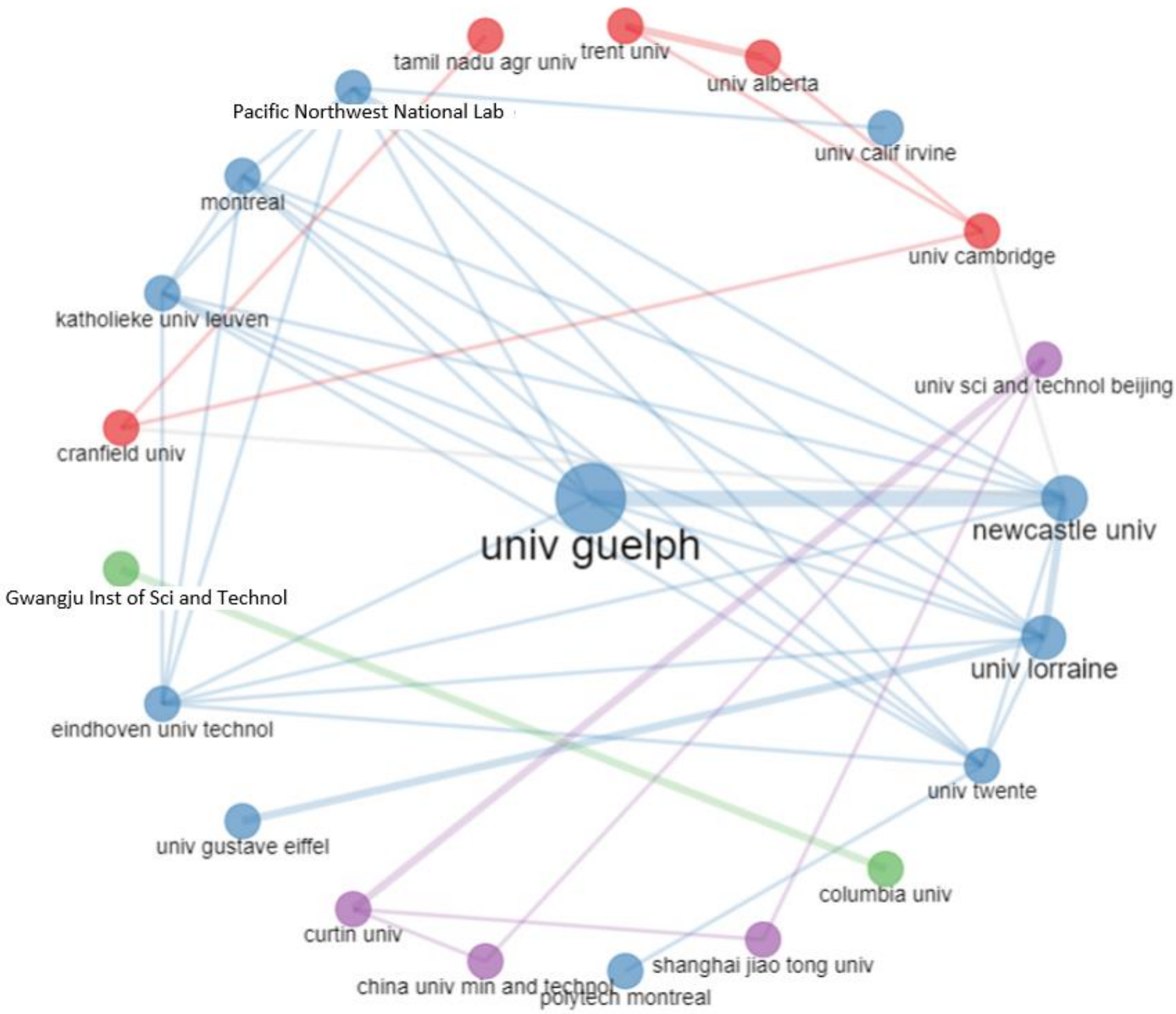


**Fig. S7.** The collaboration network of the institutions with multi-author citing articles (including self-citations). Data source: WoS (August 6th 2023).

**Sustainable Development Goals (SDGs)**

A new feature of WoS is the classification of published articles by the United Nations SDGs. **Table S3** provides the ranking of all citing articles (including self-citations). SGD 13 (Climate Action) leads the list, which is in agreement with the primary goal of mineral carbonation and waste valorization research to reduce the impact of anthropogenic activities by reducing emissions and conserving natural resources via a circular economy. This is further evidenced by the next three SDGs on the list relating to human health, consumerism, and water protection.

**Table S3.** Ranking of SDGs (classified by WoS) of the 157 citing articles (including self-citations). Data source: WoS (August 15th 2023).

| **Sustainable Development Goals** | **Record Count** |
|---|---|
| 13 Climate Action | 54 |
| 03 Good Health And Well Being | 16 |
| 12 Responsible Consumption And Production | 15 |
| 06 Clean Water And Sanitation | 9 |
| 04 Quality Education | 4 |
| 11 Sustainable Cities And Communities | 4 |
| 02 Zero Hunger | 3 |
| 07 Affordable And Clean Energy | 3 |
| 14 Life Below Water | 2 |
| 10 Reduced Inequality | 1 |
| 15 Life On Land | 1 |

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