



1<sup>st</sup> RILEM International  
Conference on  
**Mineral Carbonation for  
Cement and Concrete**

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16 and 17<sup>th</sup> April 2024

RWTH Aachen University





Dear participant of the **1<sup>st</sup> RILEM Conference on Mineral Carbonation for Cement and Concrete**,

Enforced carbonation is a relatively new but fast-growing field, as indicated by the overwhelming interest this conference has attracted. From all excellent contributions submitted we have prepared an inspiring program with 4 selected keynotes, more than 45 presentations and a dozen of posters, all packed in 2 intense days. We would like to thank all the authors for their contributions. From the recovery of waste streams, to a deep dive in carbonation mechanisms, and through an overview on the opportunities for the cement and concrete sector, we aim to cover all main topics related to mineral carbonation of cement and concrete. We hope that the discussion emerging from these contributions will lead to lasting collaborations and further projects on this exciting topic, instrumental to reach the goals of the 2050 net-zero CO<sub>2</sub> roadmaps of the cement and concrete industry.

This conference emerged from several projects ongoing at the host institute, the Institute of Building Materials Research, RWTH Aachen University. The event is part of the dissemination and co-learning activities of the Era-Min project CO2Treat, and the Horizon Europe Carbon4Minerals project, both coordinated by Dr. Liesbeth Horckmans (VITO). It is also part of the activities of the RILEM Technical committee 309-MCP “Accelerated Mineral Carbonation for the production of construction materials”, chaired by Prof. Ruben Snellings (KU Leuven), with deputy chair Prof. Thomas Matschei (RWTH University Aachen).

We are delighted to welcome you to the city of Aachen, and hope you will enjoy your stay and the conference.

The conference chairman,

**Prof. Thomas Matschei (RWTH Aachen University)**



The organization committee,

Dr. Fabien Georget (RWTH Aachen University)

Dr. Liesbeth Horckmans (VITO)

Prof. Ruben Snellings (KU Leuven)

Dr. Maciej Zajac (Heidelberg Materials)



The main aim of the **ERA-MIN 3 CO2TREAT project (2022-2025)** is to **design resource-efficient, low-carbon binder products** for durable concrete and civil engineering applications by partially substituting Portland cement with **secondary resources benefited by treatment with CO<sub>2</sub>**. Specifically, the project aims to **beneficiate 3 high-volume, alkaline industrial residues** (BOF steel slag, lignite fly ash, co-combustion ash) by CO<sub>2</sub> treatment to an intermediate product usable as **supplementary cementitious material (SCM)** in cement and concrete. These SCMs substitute virgin raw materials (limestone), secondary resources under supply pressure (hard coal combustion fly ashes, blastfurnace slag), and Portland cement.

The CO<sub>2</sub>-treated materials will be used to design sustainable binders that comply to stringent performance and durability standards for cement, concrete and civil works. Quantification of the sustainability of the use of the CO<sub>2</sub>-beneficiated materials in low-carbon cement, concrete and civil works will be done by a Techno-Economic Analysis (TEA) and Life Cycle Analysis (LCA).

The CO2TREAT project anticipates to open new recycling opportunities for large volumes of alkaline mineral residues by CO<sub>2</sub> beneficiation. Compared to the annual European cement production of 170 Mt including 38 Mt of SCMs, CO2TREAT, if realized to its maximal extent, could almost double the amount of SCMs available for composite cement production. The CO<sub>2</sub> beneficiation processes proposed by CO2TREAT would enable an additional CO<sub>2</sub> sequestration of 50-200 kg/t steel slag or 25-90 kg/t lignite and co-combustion fly ashes, or up to 2.7 Mt/y of CO<sub>2</sub> bound into stable mineral carbonates.

The CO2TREAT project is a collaboration between 5 partners from 3 countries.



CO2TREAT was funded by the ERA-MIN3 research and innovation programme on raw materials to foster the circular economy (ID:196). ERA-MIN3 has received funding from the European Union's Horizon 2020 Research and Innovation Programme under grant agreement No. 101003575. National funding was obtained from the VLAIO/Hermesfonds (Flanders), BMBF (Germany) and the Slovenian ministry of higher education, science and innovation (Slovenia).

<https://co2treat.vito.be/en>



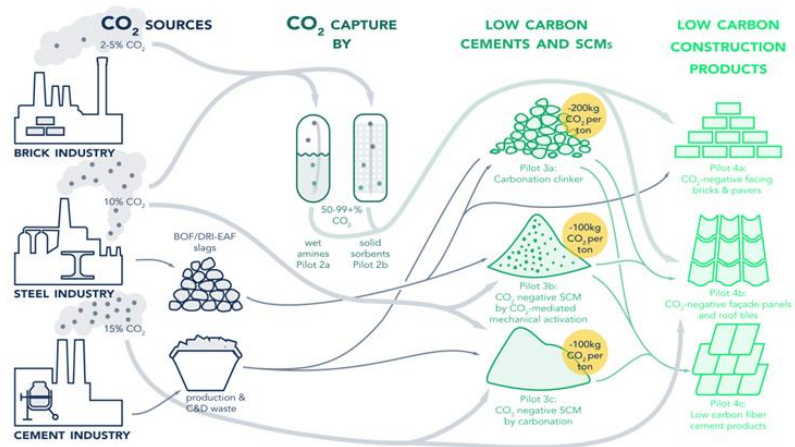


## Transforming CO<sub>2</sub> into added-value construction products

The Carbon4Minerals project is co-funded by the European Union’s Horizon Europe research and innovation program under Grant Agreement No. 101091870, and by the Swiss State Secretariat for Education, Research, and Innovation (SERI). The project commenced in January 2023.

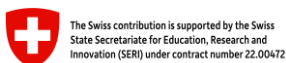
The European Green Deal has set ambitious targets for GHG emission reductions for the process industry, including steel and cement production. While transitioning to renewable energy can alleviate a significant part of GHG emissions, it offers no solution for process-inherent emissions. Residual, hard-to-abate CO<sub>2</sub> emissions from industrial processes will need to be captured, processed, and recycled into new products to meet these targets. The Carbon4Minerals project aims to address this challenge.

The core concept of Carbon4Minerals is to address the simultaneous use of CO<sub>2</sub> from industrial flue gases with current and future waste streams to unlock a vast stock of resources for innovative low-carbon binders and construction materials. This cross-sectorial innovation has the potential to reduce European CO<sub>2</sub> emissions by 46 Mt/y, equal to 10% of the EU process industry emissions, while safeguarding the competitiveness of the European industry.



The cement industry generates 6-8% of global GHG emissions and seeks alternatives to Portland clinker to reduce CO<sub>2</sub> emitted during limestone calcination. Carbon4Minerals develops innovative technologies for CO<sub>2</sub> capture and use in the production of carbon-negative minerals for high-value construction products, with the potential to reduce CO<sub>2</sub> emissions by 80%- 135% compared to cement-based reference materials. The project addresses challenges arising from dwindling supplies of low-carbon resources for cement production, while reducing emissions by 10% and safeguarding competitiveness.

The research consortium made of 14 partners from 7 different European countries will work on the concept until the end of the project, which is set for the 31st of December in 2026.



[www.carbon4minerals.eu](http://www.carbon4minerals.eu)



@carbon4minerals



**RILEM, The International Union of Laboratories and Experts in Construction Materials, Systems, and Structures, established in June 1947 in Paris, France, aims to promote scientific cooperation in the area of construction materials and structures worldwide. Presently, the association boasts 119 corporate members and over 3200 individual and staff members from 90 countries, with half actively engaged in one or more of its 46 Technical Committees.**

The mission of the association is to advance free-access scientific knowledge related to construction materials, systems and structures and to encourage transfer and application of this knowledge world-wide. RILEM's foundation rests upon its Technical Committees (TCs), comprising experts from diverse backgrounds such as academia, industry, or research laboratories, collaborating on specific subjects. TCs typically operate for 5 to 7 years and produce technical exchanges, state-of-the-art reports, and recommendations. Currently, RILEM hosts 46 active Technical Committees organized into 6 Clusters, with 4 focusing on cementitious materials, 1 on masonry, timber and cultural heritage, and 1 on polymers and asphalts.

**RILEM is also renowned for its two prestigious journals.**

***Materials and Structures***, the flagship publication of RILEM, provides a unique international and interdisciplinary forum for new research findings on the performance of construction materials. The journal, published by Springer, is dedicated to the publication of high-quality papers examining the fundamental properties of building materials, their characterization and processing techniques, modeling, standardization of test methods, and the application of research results in building and civil engineering.

***RILEM Technical Letters*** is an open access journal devoted to disseminating key breakthroughs and current contributions in the field of construction materials science. The journal focuses on the publication of short reports of major innovative research or strategic research needs in the field of construction materials and structures. By providing a fast-publishing process, *RILEM Technical Letters* constitutes a new landmark among the publications devoted to construction materials science. Publication costs are sponsored by RILEM for RILEM members.

**RILEM has been organizing symposia and workshops since its foundation.** Through its subsidiary company, RILEM Publications Sarl, RILEM has published almost 140 proceedings since 1997 and in collaboration with Springer 50 more proceedings. To broaden the education of both PhD students and the professional community, RILEM sponsors interesting and informative PhD courses and seminars on subjects of relevance to researchers working in specific areas.

**Each year, RILEM organizes two main events**, one in Spring which usually takes place in Europe, and the RILEM Annual Week in late August or early September outside of Europe. A major international conference is combined with numerous meetings of RILEM Technical Committees and Standing Committees.

**RILEM members enjoy a range of benefits, including the opportunity to propose the establishment of new Technical Committees, publish selected articles as free open-access papers in both journals, and subscribe for free to the online version of Materials and Structures. Discover more about RILEM and explore all the membership perks [here](#).**

More info : <https://www.rilem.net/>

Contact : <https://www.rilem.net/contact>

## Keynote speakers

**Dr Célia Sapart**

CO2 Value Europe  
([co2value.eu](http://co2value.eu))



**Dr. Maciej Zajac**

Heidelberg Materials  
([www.heidelbergmaterials.com](http://www.heidelbergmaterials.com))



**Prof. Zichao Liu**

Wuhan University of Technology  
([english.whut.edu.cn/](http://english.whut.edu.cn/))



**Prof. Chi-sun Poon**

The Hong-Kong Polytechnique University  
([www.polyu.edu.hk/cee/~cecspon/](http://www.polyu.edu.hk/cee/~cecspon/))



**Tuesday 16th April 2024**

<b>Welcome session</b>		
Tuesday, 16.4.	09:00 - 09:15	SuperC - main room
<b>1</b>	<b>Welcome speech</b> Matschei, Thomas <i>Institute of Building Materials Research, RWTH Aachen University, Aachen, Germany</i>	
<b>2</b>	<b>CO<sub>2</sub>Treat and Carbon4Minerals</b> Horckmans, Liesbeth <i>Vlaamse Instelling voor Technologisch Onderzoek (VITO), Mol 2400, Belgium,</i>	
<b>3</b>	<b>RILEM Presentation</b> Snellings, Ruben <i>KU Leuven, Belgium</i>	

<b>Celia Sapart: Perspective on CCU in Europe</b>		
Tuesday, 16.4.	09:15 - 09:45	SuperC - main room
Chairman	<b>Thomas Matschei (RWTH)</b>	

<b>Recycling (1): Upscaling and industrial perspectives</b>		
Tuesday, 16.4.	09:45 - 10:30	SuperC - main room
Chairman	<b>Thomas Matschei (RWTH)</b>	
<b>1</b>	<b>Use of carbonated recycled aggregate and concrete slurry waste in recycling concrete – results of the Swiss "DemoUpCarma"-project</b> Leemann, Andreas (1,2); Winnefeld, Frank (1); Münch, Beat (1); Tiefenthaler, Johannes (3) <i>1: Empa, Swiss Federal Laboratories for Materials Science and Technology, Laboratory for Concrete and Asphalt, 8600 Dübendorf, Switzerland; 2: School of Geography and Environmental Sciences, Ulster University, Coleraine, UK; 3: Neustark AG, Freiburgstrasse 251, 3018 Bern, Switzerland</i>	
<b>2</b>	<b>Industrial experiences of enforced carbonation of recycled concrete paste</b> Pato, Nicolas; Hanafi, Ahmed; Chen, Yen-Hau; Skocek, Jan; Zajac, Maciej <i>Heidelberg Materials AG, Germany</i>	
<b>3</b>	<b>Carbonated Construction Demolition Material based on concrete sludge as new Supplementary Cementitious Material in cement</b> MORIN, Vincent; MEYER, Vincent; HUET, Bruno; GAGEY, Fabrice <i>Holcim Innovation Center, France</i>	

<b>Coffee break</b>		
Tuesday, 16.4.	10:30 - 11:00	SuperC - foyer

<b>Carbonation Mechanisms (1)</b>		
Tuesday, 16.4.	11:00 - 12:30	SuperC - main room
Chairman	<b>Nishant Garg (University of Illinois)</b>	
<b>1</b>	<b>Growth of calcium carbonate in recycled cement paste induced by different accelerated carbonation regimes: the role of water</b> MA, ZIHAN; CAI, Yamei; JIANG, Yi; SHEN, Peiliang; POON, Chi sun <i>The Hong Kong Polytechnic University, Hong Kong S.A.R. (China)</i>	
<b>2</b>	<b>CO<sub>2</sub> sequestration by recycled concrete aggregates: impact of temperature and water content on carbonation</b>	



CORVEC, Gaël (1,2); Turcry, Philippe (1); Ait-Mokhtar, Abdelkarim (1); Artoni, Riccardo (2); Richard, Patrick (2)  
 1: LaSIE, UMR CNRS 7356, La Rochelle Université, 17000 La Rochelle, France; 2: Université Gustave Eiffel, MAST-GPEM, 44344 Bouguenais, France

**3 Quantification and stability of hydrous carbonate sites in calcite formed by aqueous carbonation of cement pastes**

Maia Neto, Fabio; Skibsted, Jørgen  
 Department of Chemistry and Interdisciplinary Nanoscience Center (iNANO), Aarhus University, Denmark

**4 Effect of carbonation on cementitious materials microstructure by X ray tomography**

EL FAQIR, Chakib (1,2); TENGATTINI, Alessandro (1,2); HUET, Bruno (3); MORIN, Vincent (3); BRIFFAUT, Matthieu (4); DAL PONT, Stefano (2)  
 1: Institut Laue Langevin, France; 2: Univ. Grenoble Alpes, CNRS, Grenoble INP, 3SR, 38000 Grenoble, France; 3: Holcim Innovation Center, 95 Rue du Montmurier, 38070 Saint-Quentin-Fallavier, France; 4: CNRS, Centrale Lille, UMR9013-LaMcube-Laboratoire de mécanique multiphysique et multiéchelle, Université de Lille, F-59000, Lille, France

**5 CO<sub>2</sub>-induced Mineralogical Changes in Concrete Wash Water: Thermodynamic and Experimental Assessments**

Sargam, Yogiraj (1); Hanmore, Alex (1); Matschei, Thomas (2); Georget, Fabien (2); Monkman, Sean (3)  
 1: CarbonCure Technologies, Canada; 2: RWTH Aachen University, Germany; 3: ICON, USA

**6 Mineral carbonation of construction and demolition waste sludge for CO<sub>2</sub> sequestration**

Charitha, Vootukuri; Sharma, Meenakshi  
 Department of Civil Engineering, Indian Institute of Technology Hyderabad, India

**Lunch**

Tuesday, 16.4. 12:30 - 13:30 SuperC - foyer

**Maciej Zajac: Application of Carbonation Technologies to the Cement and Concrete Industry**

Tuesday, 16.4. 13:30 - 14:00 SuperC - main room  
 Chairman **Ruben Snellings (KU Leuven)**

**Carbonation Mechanisms (2)**

Tuesday, 16.4. 14:00 - 15:00 SuperC - main room  
 Chairman **Ruben Snellings (KU Leuven)**

**1 CO<sub>2</sub> mineralization of hardened cement pastes: on the influence of cement composition on CO<sub>2</sub> uptake and pozzolanic reactivity**

Bader, Tobias; Pesch, Katja; Böhm, Matthias; Rickert, Jörg  
 VDZ Technology gGmbH, Germany

**2 Carbonation of Different Hydrated Cement Pastes – A 29Si and 27Al NMR study**

Deffner, Lukas (1); Singer, Marie (2); Jansen, Daniel (2); Gädt, Torben (1)  
 1: Technical University Munich, Germany; 2: Friedrich Alexander University Erlangen-Nürnberg

**3 Effects of Relative Humidity and Temperature on Carbonation Dynamics of CaO and Ca(OH)<sub>2</sub>**

Sun, Lisa Mingzhe (1); Hewson, Connor (1); Naderi, Majid (1); Williams, Daryl (1,2); Iacomì, Paul (1)  
 1: Surface Measurement Systems Ltd., Alperton, London, HA0 4PE; 2: Department of Chemical Engineering, Imperial College London, SW7 2AZ

**4 Composition and thermodynamic properties of the alumina-silica gel formed upon aqueous carbonation of C-S-H and ettringite**

Song, Jiayi (1); Zajac, Maciej (2); Lothenbach, Barbara (3); Skibsted, Jørgen (1)  
 1: Department of Chemistry and Interdisciplinary Nanoscience Center (iNANO), Aarhus University, Denmark; 2: Global R&D HeidelbergCement AG, Germany; 3: Empa, Laboratory for Concrete & Construction Chemistry, Switzerland

**Mineralization (1): giving a hand to carbonation**

Tuesday, 16.4. 14:00 - 15:00 SuperC - second room  
 Chairman **Torben Gädt** (TUM School of Natural Sciences (NAT))

- 1 Advancing mineral carbonation in Cement-Based Materials through Tailored Amine Blends**  
 Elakneswaran, Yogarajah (1); Kopitha, Kirushnapillai (1); Iresha, Harshani (1); Kitagaki, Ryoma (1); Saito, Ryosuke (2); Yano, Keiichi (2); Yoda, Yuya (2); Tsujino, Masato (2); Nishida, Akira (2); Senboku, Hisanori (1)  
*1: Hokkaido University, Japan; 2: Shimizu Corporation*

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- 2 Accelerating CaCO<sub>3</sub> Precipitation of Recycled Concrete Aggregates through Enzyme Carbonic Anhydrase**  
 Chen, Xiulin (1); Zhang, Zhidong (1); Giovanoli, Diego (2); Angst, Ueli (1)  
*1: Durability of Engineering Materials, ETH Zürich, Switzerland; 2: Complex Materials, ETH Zürich, Switzerland*

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- 3 Effect of triethanolamine on carbonation of clinker-free Class C fly ash**  
 Lee, Jihoon (1); Lim, Ahyun (1); Jeon, Dongho (2); Moon, Juhyuk (1,3)  
*1: Seoul National university, Republic of Korea (South Korea); 2: Dong-A University, Republic of Korea (South Korea); 3: Institute of Construction and Environmental Engineering, Republic of Korea (South Korea)*

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- 4**

**Coffee break**

Tuesday, 16.4. 15:00 - 15:30 SuperC - foyer

**Measuring carbonation and LCA (1): Measuring the carbonation degree**

Tuesday, 16.4. 15:30 - 17:00 SuperC - main room  
 Chairman **Maciej Zajac** (Heidelberg Materials)

- 1 Comparative studies on the methods to evaluate carbonation degree and activity of calcium-rich solids**  
 Zhang, Qi; Feng, Pan  
*Southeast University, China, People's Republic of*

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- 2 Collaborative laboratory experiments to measure the rate of CO<sub>2</sub> captured after accelerated carbonation by a calcimetric method**  
 Braymand, Sandrine (1); Roux, Sébastien (2); Vial, Virginie (3); Bertola, Julie (3); Cazacliu, Bogdan (4)  
*1: ICube Université de Strasbourg, France; 2: IJL, Université de Lorraine, France; 3: Vicat L'Isle d'Abeau, France; 4: GPEM, Université Gustav Eiffel, France*

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- 3 Principle of a new method to determine the CO<sub>2</sub> binding capacity of recycled concrete aggregates**  
 Turcry, Philippe (1); Hou, Yunlu (2,3); Corvec, Gaël (1,4); Mahieux, Pierre-Yves (1); Lux, Jérôme (1); Aït-Mokhtar, Abdelkarim (1); Lau Hiu Hoong, Jean David (5)  
*1: La Rochelle Université, CNRS, France; 2: State Key Laboratory of Geomechanics and Geotechnical Engineering, Institute of Rock and Soil Mechanics, Chinese Academy of Sciences, Wuhan 430071, China; 3: University of Chinese Academy of Sciences, Beijing 10*

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- 4 Tracking and Quantifying the Extent of Carbonation via Raman Imaging**  
 RATHNA KUMAR, SUDHARSAN; GARG, NISHANT  
*University of Illinois Urbana Champaign, United States of America*

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- 5 Advancing Carbonation Tracking: The Pivotal Role of Carbon Isotope Analysis in Modern Techniques**  
 Jansen, Daniel; Singer, Marie  
*Friedrich Alexander Universität Erlangen Nürnberg, Germany*

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- 6 Assessment of methods to determine the carbonation degree of crushed concrete for recycled aggregates**  
 Canul Polanco, Jennifer Anette; Sigvardsen, Nina Marie; Jakobsen, Ulla Hjorth; Ebert, Benjamin Alexander Regaard  
*Danish Technological Institute (DTI), Taastrup, Denmark*

## Mineralization (2): Industrial wastes

Tuesday, 16.4. 15:30 - 17:00 SuperC - second room

Chairman **Wilson Ricardo Leal da Silva (FLSmidth)**

- 1 CO2 mineralisation of fresh mixture to produce cement and BOF steel slag pastes**  
 Ferrara, Giuseppe (1); Humbert, Pedro (2); Nispel, Michael (3); Garufi, Davide (2); Palmero, Paola (1)  
*1: Politecnico di Torino, INSTM R.U. PoliTO-LINCE, Department of Applied Science and Technology, Corso Duca Degli Abruzzi 24, 10129 Torino (TO), Italy.; 2: Innovation Centre for Sustainable Construction, CRH, De Klencke 10, 1083 HL, Amsterdam, Netherland; 3: Fels Vertriebs und Service GmbH & Co. KG, Geheimrat-Ebert-Str. 12, 38640 Goslar, Germany*

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- 2 Kinetic study on fly ash mineral carbonation**  
 Bolourchian Tabrizi, Seyed Zuhair (1,2); Leal da Silva, Wilson Ricardo (1); Barbera, Elena (2); Bezzo, Fabrizio (2)  
*1: FLSmidth Cement, Green Innovation, Denmark; 2: Department of Industrial Engineering, University of Padova, via Marzolo 9, 35131 Padova PD, Italy*

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- 3 Transforming lignite fly ash into a carbon neutral SCM through mineral carbonation**  
 Felten, Christian Claus; Kruppa, Henning; Vollpracht, Anya; Matschei, Thomas  
*RWTH Aachen University, Germany*

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- 4 Carbonation of electric arc furnace steel slag under hydrothermal condition**  
 Huang, Liming; Babaahmadi, Arezou  
*Chalmers university of technology, Sweden*

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- 5 Carbonation of crystalline blast furnace and electric arc furnace slags**  
 Blotevogel, Simon (1,2); Saldi, Giuseppe (3,4); Castillo, Alain (3); Benezeth, Pascale (3); Patapy, Cedric (2); Cyr, Martin (2)  
*1: Ecozem Materials, 324061, Block F1, Eastpoint Business Park, Dublin 3, Ireland.; 2: LMDC, Université de Toulouse, INSA/UPS Génie Civil, Toulouse, France; 3: Géosciences Environnement Toulouse (GET), Observatoire Midi Pyrénées, Université de Toulouse, C*

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- 6 Production of cement replacement (SCM) by CO2-beneficiation of steel slag**  
 Horckmans, Liesbeth (1); Kazemi Kamyab, Hadi (1); Nielsen, Peter (1); Riems, Bert (2)  
*1: Vlaamse Instelling voor Technologisch Onderzoek (VITO), Mol 2400, Belgium.; 2: Arcelor Mittal, John Kennedylaan 51,9042 Gent, Belgium*

## Poster session

Tuesday, 16.4. 17:00 - 18:00 SuperC - second room

- 1 Enhanced weathering of returned concrete: constraining CO2 uptake by carbonation for baseline vs intervention**  
 Rateau, Rémi (1); Magee, Ruadhán (1); Žurovec, Ognjen (1); Ní Mhaoldomhnaigh, Ciara (1); Bryson, Maurice (1); McDermott, Frank (2)  
*1: Silicate Carbon Limited, NovaUCD, Dublin, Ireland.; 2: University College Dublin, UCD School of Earth Sciences, Dublin, Ireland*

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- 2 UNDERSTANDING CALCIUM CARBONATE CRYSTALLIZATION PATHWAYS FOR CONCRETE RECYCLING**  
 Dehue, Mathilde (1); Magnin, Valérie (2); Lanson, Martine (3); Ben Haha, Moshen (4); Zajac, Maciej (5); Fernandez-Martinez, Alejandro (6); Hazemann, Jean-Louis (7)  
*1: ISTERre, France; 2: ISTERre, France; 3: ISTERre, France; 4: HeidelbergMaterials; 5: HeidelbergMaterials; 6: ISTERre, France; 7: NEEL, France*

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- 3 Effect of the composition of well-hydrated slag cement pastes on their reactivity as SCMs after enforced carbonation**  
 Machner, Alisa; Schnürer, Luis  
*Technical University of Munich (TUM), Germany*

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- 4 Understanding how organic molecules control portlandite nucleation at the nanoscale**  
 Baken, Annet (1,2,3,4); Van Driessche, Alexander E.S. (5); Fernandez-Martinez, Alejandro (2,3,4); Kellermeier, Matthias (6); di Michiel, Marco (1); Lanson, Martine (2,3)  
*1: ESRF; 2: ISTERre; 3: Univ. Grenoble-Alpes; 4: CNRS; 5: IACT CSIC-UGR; 6: BASF SE*

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- 5 Concrete carbon mixing: A potential CO2 sink**  
 Davolio, Marco (1); Gelain, Stefano (2); Furlani, Federico (2); Muciaccia, Giovanni (1); Ferrara, Liberato (1)  
*1: Department of Civil and Environmental Engineering, Politecnico di Milano, Italy; 2: Simem S.p.A.*

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- 6 pH-buffering limitations on aqueous carbonation of wollastonite**

Allah, Prince; Illikainen, Mirja; Kinnunen, Paivo; Perumal, Priyadharshini  
*University of Oulu, Finland*

**7 Aqueous Carbonation of Recycled Concrete Fines: Towards Higher Efficiency**

Cai, Yamei; Jiang, Yi; Shen, Peiliang; Poon, Chi Sun  
*The Hong Kong Polytechnic University, Hong Kong S.A.R. (China)*

**8 Progress of NEDO Moonshot Project “Calcium Carbonate Circulation System in Construction”**

Noguchi, Takafumi (1); Maruyama, Ippei (1); Kitagaki, Ryoma (2); Kanematsu, Manabu (3); Tamura, Masaki (4); Fujimoto, Satoshi (5); Tsujino, Masato (6); Nakazawa, Haruo (6); Kuroda, Yasuhiro (6); Hyodo, Hikotsugu (7); Hirao, Hirohi (7); Masuo, Takayoshi (8)  
*1: The University of Tokyo, Japan; 2: Hokkaido University, Japan; 3: Tokyo University of Science, Japan; 4: Kogakuin University, Japan; 5: Utsunomiya University; 6: Shimizu Corporation; 7: Taiheiyō Cement; 8: Masuo Recycle*

**9 Co reactive: Olivine Based CO<sub>2</sub> Mineralization for Sustainable Supplementary Cementitious Materials**

Bremen, Andreas (1,2); Kleineberg, Orlando (1,2); Skylaruk, Jan (1,2)  
*1: Process Systems Engineering (AVT.SVT), RWTH Aachen University, 52074 Aachen, Germany; 2: Co-reactive GbR, 52074 Aachen, Germany*

**10 CO<sub>2</sub> Mineralization of Red Mud: Effect of CO<sub>2</sub> Pressure and Temperature**

Duraisamy, Saranyadevi; Chaunsali, Piyush  
*Indian Institute of Technology Madras, India*

**11 Investigation of Sample Preparation and Quantification of CO<sub>2</sub> Sequestered Recycled Concrete Aggregates**

Oliva Rivera, Alexander (1,2); Srivastava, Sumit (1); Mortensen, Gry Møl (1); Suchorzewski, Jan (1); Atongka Tchoffor, Placid (1); Malaga, Katarina (1,2)  
*1: RISE Research Institutes of Sweden, Dept. Infrastructure and Concrete Technology, Sweden; 2: University of Borås, Dept. Resource Recovery and Building Technology, Sweden*

**12 Combined pozzolanic and hydraulic SCMs, the case of carbonated industrial waste**

Caprai, Veronica (1); Georget, Fabien (2); Belli, Alberto (1); Keulen, Arno (1); Matschei, Thomas (2)  
*1: CRH BV, Netherlands, The; 2: Institute of Building Materials Research, RWTH Aachen University, Germany*

**13 Valorization of mineral waste through accelerated carbonation**

Grigoletto, Sophie (1); Kameni Wontcheu, Armel Florent (1); Aubry, Elise (2); Marquis, Séverine (3); Courard, Luc (1)  
*1: Urban and Environmental Engineering, University of Liège, Belgium; 2: Carmeuse Europe S.A., Belgium; 3: Centre Terre et Pierre, Belgium*

**14 carbonation of Portland cement and use of carbonated cement paste in Portland cement**

Singer, Marie (1); Deffner, Lukas (2); Gädt, Torben (2); Neubauer, Jürgen (1); Jansen, Daniel (1)  
*1: Friedrich Alexander Universität Erlangen-Nürnberg, Germany; 2: Technische Universität München, Germany*

**15 Determination of the CO<sub>2</sub> sequestration potential of waste ashes by direct mineral carbonation treatment**

Tominc, Sara (1); Carević, Ivana (2); Cerковиć, Sonja (2); Zimprich, Eva (2); Štirmer, Nina (2); Kirkelund, Gunvor M. (3); Ottosen, Lisbeth M. (3); Adhikary, Sumankumar (4); Perumal, Priyadharshini (4); Chen, Boyu (5); Hussein, Abdellah (5); Ye, Guang (5)  
*1: Slovenian National Building and Civil Engineering Institute, Slovenia; 2: University of Zagreb, Faculty of Civil Engineering, Croatia; 3: Technical University of Denmark, Department of Environmental and Resource Engineering, Denmark; 4: University of O*

**16 Carbonation of Mg(OH)<sub>2</sub>**

Franke, Saskia; Goetz-Neunhoeffler, Friedlinde; Neubauer, Jürgen  
*Friedrich-Alexander-Universität Erlangen-Nürnberg, Germany*

**17 The prospect of recalcification as a repair technique for carbonated cementitious material**

Nguyen, Thinh (1); Phung, Quoc Tri (2); Maes, Norbert (2); De Schutter, Geert (3); Cizer, Özlem (1)  
*1: KU Leuven, Belgium; 2: SCKCEN, Belgium; 3: Ghent University, Belgium*

**Conference dinner**

Tuesday, 16.4.

18:30 - 22:00

Ratskeller Aachen

Wednesday 17th April 2024

**Prof. Zhichao Liu: Transforming CO2 into building products: Innovations and Future Prospects in China**

Wednesday, 17.4. 08:30 - 09:00 SuperC - main room  
 Chairman **Fabien Georget** (RWTH Aachen University)

**Measuring carbonation and LCA (2): CO2 uptake**

Wednesday, 17.4. 09:00 - 10:00 SuperC - main room  
 Chairman **Philippe Turcry** (La Rochelle Université)

**1 Determination of the CO2 Uptake of Construction Products Manufactured by Mineral Carbonation**

Nielsen, Peter; Quaghebeur, Mieke  
*VITO, Sustainable Materials Management, Boeretang 200, 2400 Mol, Belgium*

**2 Quantification of inorganic carbon in cementitious materials: A report of Japanese project**

Maruyama, Ippei (1); Cheng, Luge (1); Takahashi, Haruka (2)  
*1: The University of Tokyo, Japan; 2: Taiheiyō consultant*

**3 Carbon sink role of lime-based construction units: Carbonation and time-dependant LCA study**

Sambataro, Luciano; Ukrainczyk, Neven; Koenders, Eddie  
*TU Darmstadt, Germany*

**Coffee break**

Wednesday, 17.4. 10:00 - 10:30 SuperC - foyer

**Carbonation Hardening and Curing**

Wednesday, 17.4. 10:30 - 12:00 SuperC - main room  
 Chairman **Liesbeth Horckmans** (VITO)

**1 Actively Carbonated Cellular Cement Foam**

Butler, Marko (1); Meyer, Diana (1); Felten, Christian (2); Bon, Volodymyr (3); Mechtcherine, Viktor (1); Vollpracht, Anya (2); Kaskel, Stefan (3)  
*1: Technische Universität Dresden, Institute for Construction Materials, Germany; 2: RWTH Aachen, Institute of Building Materials Research, Germany; 3: Technische Universität Dresden, Institute for Inorganic Chemistry, Germany*

**2 Assessing the suitability of Fe-rich industrial by-products for the production of carbonate-based binders**

Ellwood, Lucy Rachel  
*Sheffield University, United Kingdom*

**3 Characterizing the Pore Structure of CO2-injected Cementitious Systems**

Sargam, Yogiraj; Varga, Igor De La  
*CarbonCure Technologies, Canada*

**4 Hydration and Carbonation of Lime-cement binders in Carbonation Curing Environments**

Zeng, Yu; Ye, Guang  
*TU Delft, The Netherlands*

**5 Internal carbonation and hydration of cementless steel slag binder with the addition of sodium-based carbonates**

Park, Jiseul (1); Lim, Ahyeon (1); Kim, Seohyun (1); Moon, Juhyuk (1,2)  
*1: Department of Civil and Environmental Engineering, Seoul National University, Korea, Republic of (South Korea); 2: Institute of Construction and Environmental Engineering, Seoul National University, Korea, Republic of (South Korea)*

**6 Belite clinkers based on recycled concrete paste for carbonation hardened building products.**

Dietermann, Martina; Pato, Nicola; Zajac, Maciej

### Lunch

Wednesday, 17.4. 12:00 - 13:00 SuperC - foyer

### Prof. Chi-sun Poon: High-Efficiency Recycling of Waste Concrete Using Accelerated Carbonation Technology

Wednesday, 17.4. 13:00 - 13:30 SuperC - main room  
 Chairman **Jean Michel Torrenti (ESITC Paris)**

### Recycling (2)

Wednesday, 17.4. 13:30 - 15:00 SuperC - main room  
 Chairman **Jean Michel Torrenti (ESITC Paris)**

**1 Advanced carbonation technologies for total recycling of concrete wastes**

Shen, Peiliang; Jiang, Yi; Ma, Zihan; Poon, Chi sun  
*The Hong Kong Polytechnic University, Hong Kong S.A.R. (China)*

**2 Carbonation behaviour of recycled fines derived from blended cementitious materials: composition, microstructure and mechanism**

Jiang, Yi; Sun, Keke; Shen, Peiliang; Poon, Chi-Sun  
*Department of Civil and Environmental Engineering & Research Centre for Resources Engineering towards Carbon Neutrality, The Hong Kong Polytechnic University, Hong Kong, China.*

**3 Investigation into using CCU material by-produced from waste concrete sludge in concrete**

WATANABE, Kenzo (1); TORICHIGAI, Takeshi (1); MORI, Kanako (1); AVADH, Kumar (1); YAGI, Toshiyuki (2); SASAKI, Takeshi (2)  
*1: KAJIMA Corporaiton, Japan; 2: Nippon Concrete Industries Co., Ltd, Japan*

**4 Investigation of Sample Preparation and Quantification of CO<sub>2</sub> Sequestered Recycled Concrete Aggregates**

Oliva Rivera, Alexander (1,2); Srivastava, Sumit (1); Mortensen, Gry Møl (1); Suchorzewski, Jan (1); Atongka Tchoffor, Placid (1); Malaga, Katarina (1,2)  
*1: RISE Research Institutes of Sweden, Dept. Infrastructure and Concrete Technology, Sweden; 2: University of Borås, Dept. Resource Recovery and Building Technology, Sweden*

### Mineralization (3)

Wednesday, 17.4. 13:30 - 15:00 SuperC - second room  
 Chairman **Frank Winnefeld (Empa)**

**1 Exploring the Potential of Mineral Carbonation in Cement Production with Carbonated SCMs**

Leal da Silva, Wilson Ricardo; E. Nielse, Tobias; Rasmussen, Mads; L. Williamson, David; Clausen, Kim  
*FLSmidth Cement, Denmark*

**2 Upcycling different sources of RCP into a carbon negative SCM through mineral carbonation**

Kamyab, Hadi; Nielsen, Peter  
*Vlaamse Instelling voor Technologisch Onderzoek (VITO), Mol 2400, Belgium,*

**3 Synthesis of pure vaterite via leaching-carbonation of basic oxygen furnace slag**

Song, Qifeng (1); Guo, Ming-Zhi (2); Tung-chai, Ling (1)  
*1: Hunan University, China, People's Republic of; 2: Shaoxing University, China, People's Republic of*

**4 Carbon uptake potential and reactivity of synthetic calcium magnesium aluminosilicate glasses**

Machner, Alisa (1); Panda, Subhashree (2); Schnürer, Luis (1); Ruiz Pestana, Luis (2); Suraneni, Prannoy (2)  
*1: Technical University of Munich (TUM), Germany; 2: University of Miami, USA*

### Coffee break, Poster, and Networking



# CO<sub>2</sub> mineralization of hardened cement pastes: on the influence of cement composition on CO<sub>2</sub> uptake and pozzolanic reactivity

Tobias Bader, Katja Pesch, Matthias Böhm, Jörg Rickert

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*Keywords: mineralization, pozzolanic reactivity, supplementary cementitious material, CCUS, circular economy*

The new cement standard EN 197-6:2023 defines cements with recycled concrete fines (RCF) as main constituent, which is an essential factor in achieving the overall goal of circular economy. Additionally, CO<sub>2</sub> mineralization of hardened cement pastes (HCP) in RCF is a promising carbon capture and utilization as well as storage (CCUS) approach to reduce CO<sub>2</sub> emissions, which may even improve the properties of the resulting supplementary cementitious material (SCM). However, the HCPs to be subjected to CO<sub>2</sub> mineralization are derived from different types of cement. The present study deals with systematic findings about the CO<sub>2</sub> binding capacity of HCPs based on cements with different compositions and their effects on pozzolanic reactivity.

Accelerated carbonation tests were carried out in a laboratory scale on mortars made from different cement types (acc. EN 197-1) with a water/cement ratio of 0.50 and natural siliceous sand. The mortar samples (age > 90 days) were crushed and ground to obtain analogues of recycled concrete fines. The accelerated aqueous carbonation experiments were carried out with a newly developed cold carbonation technique (CCT) in a custom-built continuously stirred reactor with pure CO<sub>2</sub> injection at a constant temperature of 5 °C. CO<sub>2</sub> uptake and pozzolanic reactivity were evaluated using a multi-analytical approach. This includes thermogravimetric analysis, isothermal calorimetry according to ASTM C 1897-20, powder XRD analysis in combination with Rietveld refinement (internal standard method) and mid-infrared spectroscopy in ATR mode.

The experiments carried out using CCT showed that the CO<sub>2</sub> mineralization of HCP leads to the formation of crystalline calcium carbonate and amorphous products from decalcified hydrate phases, irrespective of the cement composition. It was found that the estimated CO<sub>2</sub> uptake depends on the composition of the cement type used (type and amount of SCM). Reactivity tests according to ASTM C 1897-20 indicate that CO<sub>2</sub> mineralization of HCP can improve pozzolanic properties of the analogous fines, with the degree of improvement depending on the cement composition. It should be noted that the non-hydrated residual particles within the analogous fines may contribute to the assessment of pozzolanic reactivity, especially in cements containing granulated blast furnace slag.

The conducted study has shown that CO<sub>2</sub> mineralization of HCP by CCT is a promising approach to obtain value-added RCF with enhanced pozzolanic reactivity. However, RCF as a by-product of aggregate recycling from concrete demolition is complex and can be very different in composition compared to the materials used in this study. This circumstance may require an individual assessment of the maximum CO<sub>2</sub> sequestration potential of RCF and the resulting pozzolanic reactivity.



# Carbonation of crystalline blast furnace and electric arc furnace slags

Simon Blotevogel<sup>1,2</sup>, Giuseppe Saldi<sup>3,4</sup>, Alain Castillo<sup>3</sup>, Pascale Benezeth<sup>3</sup>, Cedric Patapy<sup>2</sup>, Martin Cyr<sup>2</sup>

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<sup>2</sup>*LMDC, Université de Toulouse, INSA/UPS Génie Civil, Toulouse, France*

<sup>3</sup>*Géosciences Environnement Toulouse (GET), Observatoire Midi Pyrénées, Université de Toulouse,  
CNRS, IRD, Toulouse, France*

<sup>4</sup>*University of Perugia, Department of Physics & Geology, via Pascoli 35, 06123 Perugia, Italy*

*Keywords: Electric arc furnace slag (EAF), crystalline blast-furnace slag, mineral carbonation*

Unlike granulated blast furnace slags, the crystalline nature of many steel slags largely limits their use as supplementary cementitious materials. One potential way of increasing their reactivity is mineral carbonation that will result in the production of Ca and Mg carbonates and a Si(Al)-gel with potential pozzolanic reactivity.

In this study a crystalline blast furnace slag and an electric arc furnace (EAF) slag were carbonated in a wet slurry reactor at 15 bar partial pressure of CO<sub>2</sub> and 120°C for 14 days to evaluate their carbonation potential.

The mineralogic composition of the slags and CO<sub>2</sub> uptake were determined before and after carbonation and the composition of the liquid phase was monitored during the reaction. Both slags contained significant amounts of (Ca, Mg)-(alumino)silicates. In the EAF-slag those were mainly C2S type minerals (larnite, bredigite, merwinite) with various Ca/Mg ratios (in total ~30 wt%) and around 10 wt% of the aluminosilicate gehlenite. In addition, the EAF-slag contains ~25 wt% of wüstite type Fe-oxide containing also Mg and Mn. On the other side, the crystalline blast furnace slag mainly contained melilite group aluminosilicates (60 wt%), and approximately 10 wt% of pseudowollastonite.

After carbonation, the crystalline blast furnace slag contained 8.4 wt% of CO<sub>2</sub> and the EAF slag 16.7 wt% of CO<sub>2</sub>, despite the higher alkaline earth content of the former (50 wt% and 37 wt% of CaO+MgO, respectively). This suggests that under the given conditions (Ca, Mg)-silicates are more readily carbonated than Ca-aluminosilicates. Furthermore, XRD analysis of the EAF carbonation products shows that not only Ca and Mg carbonates were formed, but also Fe containing carbonates (ankerite). Adding Fe significantly increases the pool of cations available for carbonation in EAF-slag. After carbonation the contents of larnite, bredigite, merwinite and wüstite were significantly reduced, showing that those phases were subject to carbonation. In contrast gehlenite content remained relatively constant. In the crystalline blast furnace slag only calcite was identified as carbonation product. After carbonation, the contents of melilite group endmembers (akermannite and gehleite) remained constant. Only pseudowollastonite and mixed melilite minerals appear to have been carbonated in this slag. The solution analysis showed that in both experiments, Ca and Si concentrations plateaued after 1 day, whereas Mg and Fe concentrations increased over a longer time. This suggests a need for longer reaction times to increase the solution concentration to a point at which these elements are incorporated in carbonates.

Overall, the results of the study show that Fe(II) contained in steel slags might be an essential constituent for carbonation, increasing the CO<sub>2</sub> uptake potential of the slags and stabilizing mobile Fe(II). On the contrary, the presence of Al in many minerals of crystalline blast furnace slags appears to make its carbonation more challenging.

# Kinetic study on fly ash mineral carbonation

Seyed Zuhair Bolourchian Tabrizi<sup>1,2</sup>, Wilson Ricardo Leal da Silva<sup>1</sup>, Elena Barbera<sup>2</sup>, Fabrizio Bezzo<sup>2</sup>

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Keywords: CO<sub>2</sub> Utilization; Kinetic Model; Sensitivity Analysis

CO<sub>2</sub> emissions from the cement industry present a significant environmental challenge, prompting the development of Carbon Capture, Utilization, and Storage solutions. Among these, mineral carbonation stands out, driving the development of carbonation reactors that could enable the carbonation of Supplementary Cementitious Materials. The availability of reliable mathematical models describing the key phenomena occurring in a carbonation reactor would represent a valuable tool to simulate and optimise the behaviour of the unit operation and to provide quantitative insight on its performance. However, this is not trivial, due to the complexity of the phenomena taking place and to a general scarcity of experimental data which adds difficulty in model identification. In this study, we compare the fitting performance of different possible formulations for shrinking-core kinetic models with an approach to simplify these models, making identification easier while ensuring they remain accurate enough.

Shrinking-core models are based on particle-fluid processes and formulated by considering a network of resistance in solution-diffusion-reaction pathway. They provide a mechanistic insight into the carbonation process and are evolving based on particle geometry, rate limiting steps (surface reaction and film / product layer diffusion), and diffusion term (reaction time correlated, and non-Fickian diffusion). We used published kinetic data for wood combustion fly ash to identify these models. Our results show that R<sup>2</sup> can increase from 0.33 to 0.99 depending on the model being used – Fig.1 shows the best model performance.

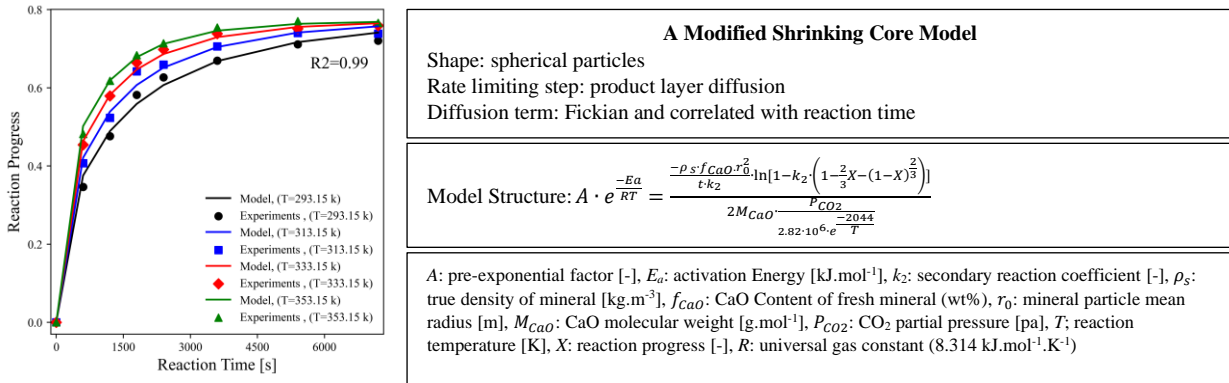


Fig.1. Reaction progress data and model behaviour for a shrinking core model.

A global sensitivity analysis (Sobol's method) on model parameters reveals the dominance of activation energy across the entire reaction field, with other kinetic parameters exhibiting minor importance, regardless the models. In particular, this analysis shows a negligible sensitivity of models to pre-exponential factor, which is consequently difficult to estimate with limited experimental data. To ease model identification, we fix this parameter to nominal values for different evolutionary models and estimate the remaining parameters. Results show that the simplified model is still able to fit data with good accuracy.

In conclusion, mineral carbonation-related kinetic studies demand reliability and cost-effectiveness due to the expense associated with kinetic data. Our study demonstrates the potential to reduce the number of parameters without compromising the model reliability, and thus to exploit the information in experimental data more effectively.

# Collaborative laboratory experiments to measure the rate of CO<sub>2</sub> captured after accelerated carbonation by a calcimetric method

Braymand S.<sup>1</sup>, Roux S.<sup>2</sup>, Vial V.<sup>3</sup>, Bertola J.<sup>3</sup>, Cazacliu B.<sup>4</sup>

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<sup>2</sup>Laboratoire IJL, Université de Lorraine, France

<sup>3</sup>Vicat L'Isle d'Abeau, France

<sup>4</sup>Laboratoire GPEM, Université Gustav Eiffel, France

*Keywords: Recycled concrete aggregate; Accelerated carbonation; Calcimetry; CO<sub>2</sub> measurement.*

The French Fastcarb project objectives included the storage of CO<sub>2</sub> in recycled concrete aggregates (RCA). As part of these, it was necessary to evaluate techniques for measuring CO<sub>2</sub> captured during accelerated carbonation. In order to assess the effectiveness of a process and determine any associated environmental benefits, an accurate assessment of the CO<sub>2</sub> mineralised during carbonation is required.

The aim of this study was to carry out collaborative experiments (lab1, lab2, lab3) to measure the rate of CO<sub>2</sub> captured after accelerated carbonation using a calcimetric method. This chemical method for quantifying CO<sub>2</sub> is based on reacting the material with an acid (HCl). It consists of using a Bernard or Dietrich-Frühling calcimeter to measure the volume released during the reaction with the acid at constant atmospheric pressure and temperature. This study was carried out on RCA (0/4 and 4/16) from the same source. These are aggregates produced from residual concrete returned to ready-mix concrete plants. A semi-industrial process was used on the site of a cement plant to achieve accelerated carbonation of RCA. A calcimetric analysis of RCA before (NC) and after carbonation process (C) was carried out in laboratories.

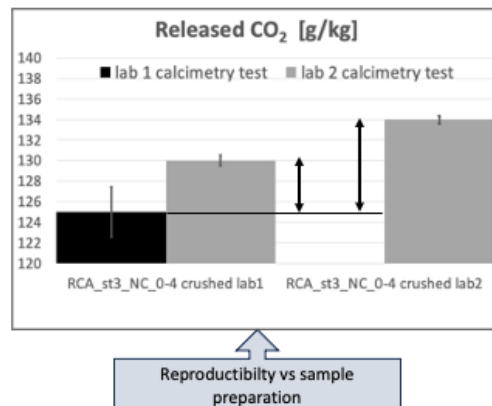
It has been shown that the calcimetric method is suitable when it is carried out in the laboratory in a controlled environment. For a same sample preparation and storage (st) the repeatability (within the same lab.) and the reproducibility (between lab.) of the measurement are very satisfactory. The repeatability of the sample preparation during the production of the powder is very satisfactory, while the reproducibility is moderate. This study contributed to the proposal of tests recommendations in the context of Fastcarb.

Inter-laboratory tests [g/kg]	Inter-laboratory mean values	Inter-laboratory standard deviation values	Average of standard deviations within laboratories
RCA_st1_NC_0-4	129.7	2.8	2.0
RCA_st1_C_0-4	147.5	8.2	3.3
RCA_st1_NC_4-16	157.0	10.7	1.9
RCA_st1_NC_4-16	154.2	7.5	2.5

Sample preparation  
in each laboratory

Repeatability

Reproducibility



# Actively Carbonated Cellular Cement Foam

Marko Butler<sup>1</sup>, Diana Meyer<sup>1</sup>, Christian Felten<sup>2</sup>, Volodymyr Bon<sup>3</sup>, Viktor Mechtcherine<sup>1</sup>, Anya Vollpracht<sup>2</sup>, Stefan Kaskel<sup>3</sup>

<sup>1</sup>*Institute for Construction Materials, TU Dresden, Dresden, Germany, marko.butler@tu-dresden.de*

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*Keywords: cellular cement foam; carbonation curing; carbonation kinetics; micro structure; mechanical properties*

Cellular foamed cements are lightweight mineral materials and can show high functional performance in terms of compressive strength and energy absorption as well as insulation. Foam concrete can be digitally processed, e.g., by 3D printing.

In the present study cellular cement foam was produced by a pre-foaming process with a bulk density of approx. 0.2 kg/L. For foams obtained from this process, the impact of active carbonation in the fresh and hardened state on mineral phases, pores structure as well as mechanical properties was investigated. For this purpose, either the protein foam was produced with CO<sub>2</sub>, so that an interaction between early carbonatable phases and CO<sub>2</sub> occurred already in the fresh state. Alternatively, CO<sub>2</sub> gas was permeated through the foam in the hardened state at various ages. In all cases, gas containing 100 % per volume of CO<sub>2</sub> was used for the experiments.

The kinetics of carbonation at different CO<sub>2</sub> concentrations was estimated using gravimetric adsorption equipment by constant gas volume and given initial gas pressure. The effect of active carbonation on the mineralogical composition of the foam was investigated using XRD measurements, TG, MIP and ESEM. The overall structure of foam was assessed by analysis of datasets resulting from  $\mu$ CT. The mechanical properties was assessed by discussion of compressive stress-displacement curves from monotonic compression tests.

The investigations showed that cement foam can be carbonated rapidly and to a high degree due to its porous structure. The addition of small volumes of biochar favors rapid carbonation by improving permeation characteristics. Depending on grain size of biochar particles, they contribute negatively or positively on strength development of foam. The inclusion of calcium carbonate depositing bacteria did not result in more favorable conditions for carbonation. In summary, the carbonation does not result in a significant improvement of the foams strength.

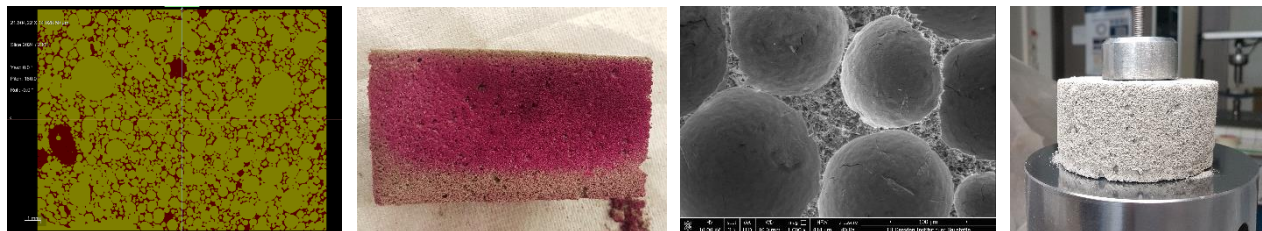


Figure (left to right): pores connectivity analysis based on CT scan, carbonation front in partly carbonated cement foam, ESEM-Micrograph of cement foam cellular pores, compression test.

# Assessment of methods to determine the carbonation degree of crushed concrete for recycled aggregates.

J.A. Canul Polanco<sup>1</sup>, N.M. Sigvardsen<sup>1</sup>, U. H. Jakobsen<sup>1</sup>, B.A.R. Ebert<sup>1</sup>

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*Keywords: Carbonation, recycled aggregates, acid dissolution, petrographic analysis, concrete waste*

Great interest in carbon uptake in concrete has emerged in recent years. The hydration products from cement can react with the CO<sub>2</sub> in the atmosphere, with calcium carbonate as the chemical reaction's final product. In connection with this, there are many tons of concrete demolition waste standing in landfills, which could be used as a CO<sub>2</sub> sink and potentially material to produce new concrete. Therefore, the development of methods to determine the degree of carbonation of concrete elements and crushed concrete particles are essential to assess the best storage and processing actions for crushed concrete between the time of demolition/crushing to the time of recycling/application in new concrete.

The objective of the study was to find a suitable method to determine the carbonation degree of crushed concrete. Three samples were evaluated: crushed mortar cast in the laboratory with known composition (reference material), crushed concrete from an industrial concrete plant exposed to carbonation due to full outdoor exposure, and the same crushed concrete without full exposure to carbonation. The carbonation degree of crushed concrete was evaluated using three methods: Selective acid dissolution combined with Ca<sup>2+</sup> titration (SACT), petrographic analysis with pH indicator, and thermal treatment.

To determine the carbonation level, it is essential to have information about the cement paste and/or the cement content. The cement paste content was determined by acid dissolution (2M HNO<sub>3</sub> solution) for methods 1 and 3. For method 2, point counting was conducted on thin sections. All methods for determining the cement paste content showed disadvantages. The primary difficulty with acid dissolution is the insoluble residue of cement and the soluble phase of the aggregates. In the case of point counting, it is challenging to obtain a representative sample and count the fine particles around the aggregates.

The carbonation degree results varied significantly depending on the method used. For crushed concrete not exposed to direct carbonation, the measured carbonation degree was 67% using method 1, 19% using method 2, and 45% using method 3. In contrast, the carbonation level of crushed concrete samples exposed to direct carbonation was 95% using method 1, 62% using method 2, and 101% using method 3. This variation could be attributed to the challenge of distinguishing the CaCO<sub>3</sub> content originating from the aggregates and that resulting from the carbonated cement paste. On the other hand, method 2 yielded the lowest carbonation degree values, possibly because the quantification of the carbonated paste is based on the areas not colored by phenolphthalein spray (a change in pH < 8.2) and not the CaCO<sub>3</sub> content directly. Consequently, results obtained by methods 1 and 3 may not be directly comparable to those obtained by method 2. Moreover, the carbonation degree determined in the non-carbonated reference sample was 24% using method 1 and 6% using method 3. This suggests that the SAM solution may not fully dissolve the CSH and Ca(OH)<sub>2</sub> phases. Additionally, the weight loss observed between 550-850°C could be linked to chemically bound water.

The authors conclude that each method has distinct limitations and recommends employing multiple methods to evaluate the carbonation degree of crushed concrete. Additional research is required to determine the reliability of using SACT to measure the carbonation degree of crushed concrete.

# Accelerating CaCO<sub>3</sub> Precipitation of Recycled Concrete Aggregates through Enzyme Carbonic Anhydrase

Xiulin Chen<sup>1</sup>, Zhidong Zhang<sup>1</sup>, Diego Giovanoli<sup>2</sup>, Ueli Angst<sup>1</sup>

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<sup>2</sup>Complex Materials, D-MATL, ETH Zürich, diego.giovanoli@mat.ethz.ch

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**Keywords:** Recycled Concrete Aggregates; Carbonic Anhydrase; CaCO<sub>3</sub> precipitation

Using recycled concrete aggregate (RCA) derived from substantial volumes of global construction waste represents an environmentally and economically friendly solution for the CO<sub>2</sub> crisis. RCA is an eco-friendly calcium source due to the presence of hydrated cement paste and exhibits potential for CO<sub>2</sub> sequestration via CaCO<sub>3</sub> precipitation. Ca<sup>2+</sup> from cement hydration products in RCA can be extracted by leaching in solutions, and subsequently, react with atmospheric CO<sub>2</sub> to form CaCO<sub>3</sub>. Nonetheless, the process of CaCO<sub>3</sub> precipitation may be impeded by various factors, with the hydration reaction of CO<sub>2</sub> serving as one of the limiting steps. To accelerate CaCO<sub>3</sub> precipitation, the application of carbonic anhydrase (CA), a class of enzymes that catalyze the hydration of CO<sub>2</sub> into H<sup>+</sup> and HCO<sub>3</sub><sup>-</sup> up to 10<sup>7</sup> times faster than the natural process, can be employed. Studies have shown that the CaCO<sub>3</sub> precipitation onset is faster with CA. Therefore, larger quantities of CaCO<sub>3</sub> can precipitate in a shorter duration with the enzyme CA.

Our research aims to achieve efficient CO<sub>2</sub> sequestration by utilizing calcium sources from RCA with the help of CA. Our preliminary results indicate that CA can significantly accelerate CO<sub>2</sub> sequestration in alkaline solutions (Fig. 1a and b), in which solutions with CA showed faster pH decreases due to the faster reaction between CO<sub>2</sub> and OH<sup>-</sup>. CA enzyme activity is essential for the efficacy of CO<sub>2</sub> sequestration. Therefore, CA enzyme activity in RCA leaching solution is investigated by using p-NPA assay. Parameter studies, covering pH, ionic strength, and different ions such as Ca<sup>2+</sup>, Mg<sup>2+</sup>, Al<sup>3+</sup>, and SO<sub>4</sub><sup>2-</sup>, are used to evaluate CA activity in RCA leaching solutions. Our preliminary results (Fig.1c) indicated that high pH is critical for CA activity, and high ionic strength of the solution has some minor effects on CA activity. The leaching scheme used in this study has a negligible effect on CA activity. The extraction of precipitates at different intervals during experiments allows diverse characterization techniques, including XRD and SEM, to elucidate the fundamental mechanisms of CA-facilitated CaCO<sub>3</sub> precipitation involving calcium leached from RCA.

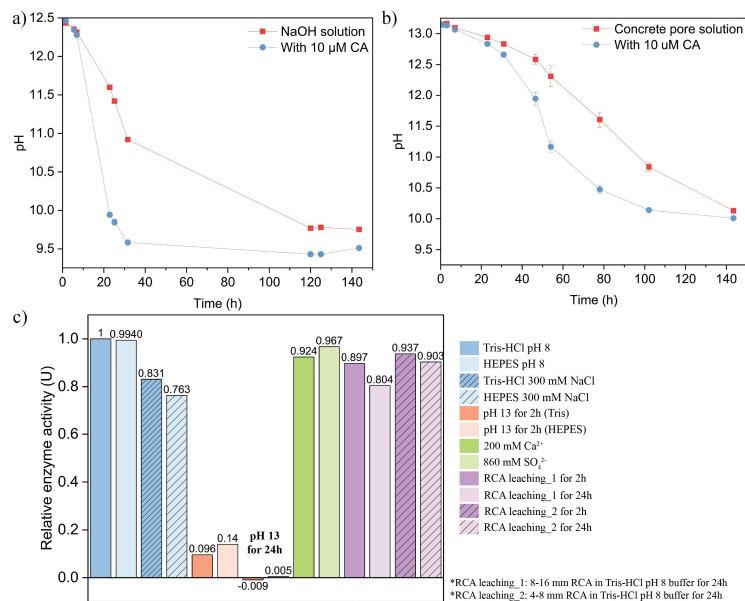


Figure 1. a) pH changes of NaOH solution with and without CA. b) pH changes of concrete pore solution with and without CA. c) CA enzyme activity under different conditions.

# An Experimental Study on the Micro-scale Deformation Behavior of CO<sub>2</sub>-Cured Di-calcium and Tri-calcium Silicate Pastes Using In-situ Loading Atomic Pair Distribution Function Analysis

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*Keywords: CO<sub>2</sub> curing, Dicalcium silicate (C<sub>2</sub>S), Tricalcium silicate (C<sub>3</sub>S), in-situ loading X-ray scattering, Pair distribution function analysis (PDF)*

Carbonation hardening of Portland cement (PC) is regarded as one of the most promising techniques for reducing the net CO<sub>2</sub> emission in the cement industry, owing to its high CO<sub>2</sub> sequestration capability. Furthermore, CaCO<sub>3</sub> crystals and modified C–S–H phases grown by the reaction between CO<sub>2</sub> and calcium silicate phases constituting PC are known to attribute to the superior mechanical properties of CO<sub>2</sub>-cured cement composites. Microstructural reinforcements by the mechanically interconnected CaCO<sub>3</sub> crystals and amorphous phases were estimated as one of the main reasons for the high performance of CO<sub>2</sub>-cured cement composites. However, the exact strength development mechanisms of reinforced microstructures need further investigation and verification. Therefore, it is imperative to investigate the microstructural deformation and load transfer mechanics of individual calcium silicate minerals to fully understand the strength development of CO<sub>2</sub>-cured cement composites. In this study, hardening characteristics and microstructural deformation behavior of the cement composites were analyzed by pure tricalcium silicate (C<sub>3</sub>S) and dicalcium silicate (C<sub>2</sub>S) pastes cured under a highly concentrated CO<sub>2</sub> environment. Phase compositions and microstructural features of the C<sub>3</sub>S and C<sub>2</sub>S pastes by curing regime were identified to clarify the microstructural phase evolution. Micro-scale compressive strength and elastic moduli were investigated to determine the hardening behavior of respective calcium silicate minerals depending on the phase evolution. As shown in Fig. 1, in-situ loading high energy X-ray scattering (XRS) experiment and pair distribution function (PDF) analysis were conducted to define the atomistic, crystallographic, and macro-scale stress-strain behavior of the CO<sub>2</sub>-cured C<sub>3</sub>S and C<sub>2</sub>S pastes. Elastic moduli and compressive strengths were determined to elucidate the hardening characteristics of calcium silicates. Comparison between the interatomic (by PDF), crystallographic (by XRS), and macro-scale (by strain gauge) deformation behavior could elucidate the load-transfer and hardening mechanisms of carbonation products of CO<sub>2</sub>-cured calcium silicate composites.

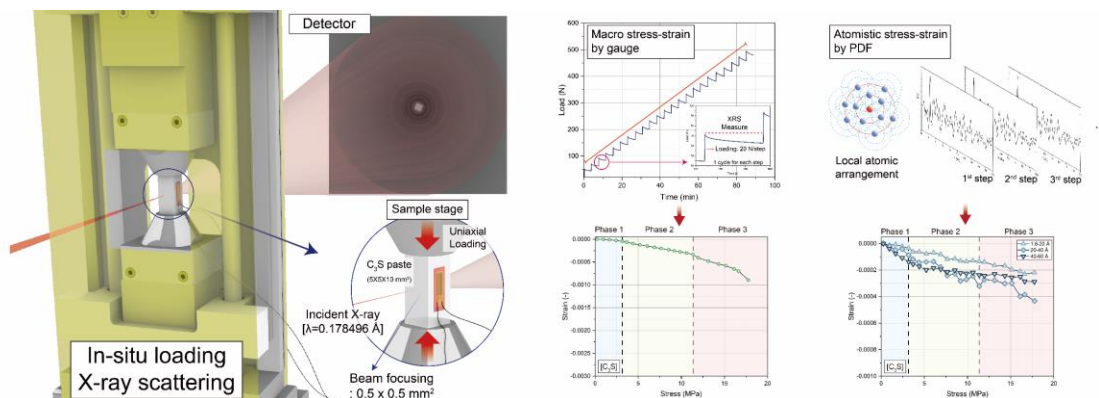


Fig. 1. A schematic diagram for in-situ loading X-ray scattering experiment.

# CO<sub>2</sub> sequestration by recycled concrete aggregates: impact of temperature and water content on carbonation

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*Keywords: Dry carbonation, CO<sub>2</sub> uptake, recycled concrete aggregates*

One strategy for reducing the carbon footprint of cementitious materials is to sequester CO<sub>2</sub> by carbonating recycled concrete aggregates (RCA) obtained from the crushing of end-of-life structures. Carbonation also has the advantage of improving the properties of the RCA such as porosity and water absorption. Gases with high CO<sub>2</sub> concentration released by cement plants could be used as reagent for the carbonation process. The temperature of these gases can reach more than 100°C and the molar fractions of CO<sub>2</sub> and water are higher than 15% and 5-10%, respectively. To develop such a process, it is necessary to understand the impact of temperature and water content on the carbonation of RCA. For this purpose, an experimental campaign was carried out on mortar discs (thickness 5mm and diameter 28mm). Firstly, samples were preconditioned at 25 or 50°C and different relative humidities (between 30 and 95%). Secondly, the specimens were carbonated in a closed cell with a 30% CO<sub>2</sub> gas at the same temperature and relative humidity than during the preconditioning step. All except one side of the samples was covered to prevent CO<sub>2</sub> penetration, so that carbonation can be considered to progress in 1D. During carbonation, the rate of CO<sub>2</sub> uptake was continuously monitored by measuring the CO<sub>2</sub> concentration in the closed cell which CO<sub>2</sub> is regularly reinjected. At the end of carbonation, bound CO<sub>2</sub> was determined by thermogravimetric analysis (TGA). The temperature and the water content of RCA has been observed to control both carbonation rate and CO<sub>2</sub> sequestration capacity (Fig. 1).

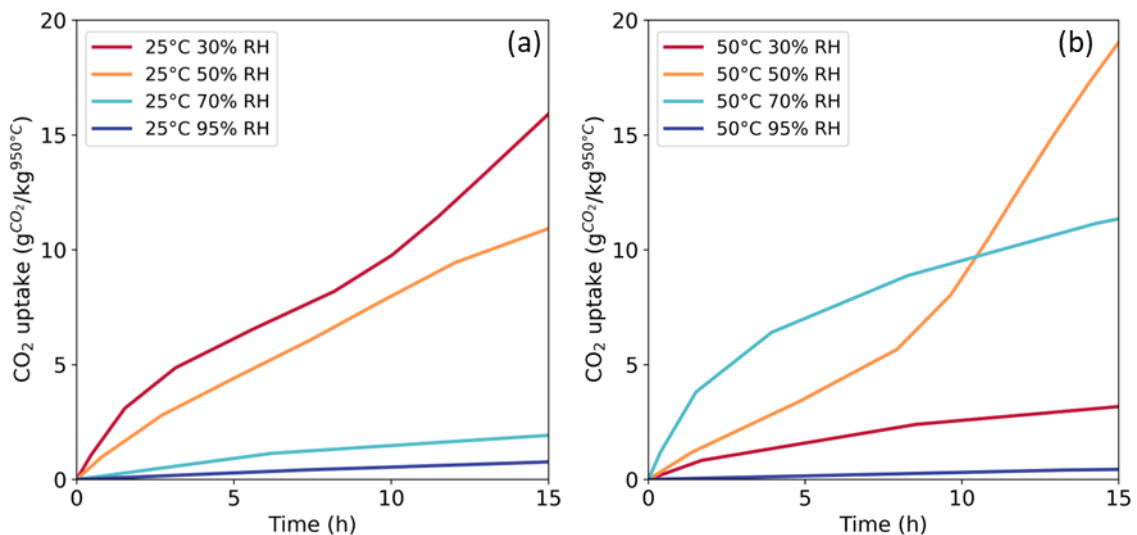


Fig. 1. Time-evolution of the CO<sub>2</sub> uptake by the studied mortar carbonated at different relative humidities at 25°C (a) and 50°C (b) (in g of CO<sub>2</sub> par kg of mortar dried at 950°C).



# Carbonation of Different Hydrated Cement Pastes – A $^{29}\text{Si}$ and $^{27}\text{Al}$ NMR study

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*Keywords: Wet-carbonation of portland cement; Solid-state NMR; Amorphous alumina-silica gel; Cement chemistry*

Carbonated recycled cement pastes (cRCP) are a new material class with potential application as supplementary cementitious materials (SCM). Wet carbonation of hydrated cement fines gives easy access to cRCPs. Their pozzolanic reactivity as SCM is attributed to the alumina-silica gel, which forms beside calcite during carbonation [1, 2]. The amorphous character of the gel impedes deeper structural characterization by quantitative X-ray diffraction (XRD) analysis. Therefore, we applied solid-state nuclear magnetic resonance (NMR) spectroscopy to study the connection between the composition of the carbonated product and the original cementitious binder chemistry, using three Portland cements (2x CEM I 42.5 R and 1x CEM I 52.5 N).

After characterization by QXRD, XRF, LDA, and BET, the three cements were hydrated for 35 days at 50°C, finely ground, and subsequently carbonated by bubbling pure CO<sub>2</sub> through a suspension of the ground, hydrated cement for 2 h. The resulting solids – hydrated and carbonated – were subjected to  $^{29}\text{Si}$  and  $^{27}\text{Al}$  solid-state NMR analysis. To quantify all silicon species present, the deconvolution approach, described by Skibsted [3] and Neto [4], was adopted.

Using the relative quantities from the deconvoluted Si-spectra, we report on the composition of the hydrated binder, e.g., the hydration degree, the average chain length of the silicate chains, and the amount of Al in the C-S-H phases. Not surprisingly, the cement with the smallest particle size distribution showed the largest degree of hydration, as determined by  $^{29}\text{Si}$  NMR. Furthermore, the peaks in the  $^{27}\text{Al}$  spectra confirm the presence of aluminum species in the CSH phase (Al<sub>IV</sub>, Al<sub>V</sub>, and Al<sub>VI</sub>), ettringite, and AFm. After carbonation, the silicate and aluminate ions are mostly found in the amorphous gel. After 2h of carbonation, the reaction is not complete, and residual AFm, CASH, and anhydrous clinker phases are also detected. Again, the cement with the greatest surface area, CEM I 52.5 N, exhibited the largest gel content. We assign the faster decalcification process of this binder to a higher hydrate phase content, initially caused by finer particles.

In summary, we describe how the raw material properties of three Portland cements and their hydration products influence the composition of the final carbonated product. Ultimately, we are interested in the influence of admixtures on the kinetics and morphology of the carbonated cement hydrates in future studies.

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# Belitic clinkers based on recycled concrete paste for carbonation hardened building products

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*Keywords: belitic cement; mineralization; carbonation hardening*

CO<sub>2</sub> mineralization, as a technology within CCUS solutions, has the potential to become an attractive decarbonization technology due to its ability to permanently store carbon. Moreover, CO<sub>2</sub> mineralization technology has the added benefits of either yielding new products or improving the performance of building materials and is achievable with lower energy demands.

This contribution reports the progress of the projects focused on developing belite clinkers for carbonation hardening applications. Belite clinkers are characterized by lower CO<sub>2</sub> emissions due to their lower clinkering temperature and lower CaO content, compared to Portland cement clinker. To further reduce these emissions, the use of Recycled Concrete Powder (RCP) to replace limestone in the raw mix formulation is being investigated. RCP, the fine fraction obtained from concrete recycling, is a source of CO<sub>2</sub>-free calcium and can further reduce the CO<sub>2</sub> footprint when replacing limestone in the formulation of belitic clinkers.

Both laboratory tests and semi-industrial production trials have demonstrated that the production of such belite clinkers is possible. Due to the variable and complex chemical and mineralogical composition of RCP, several minor phases are formed during the clinkering. However, Belite remains as the main phase with > 50 wt.% in the clinker. At the same time it was proven that clinkering in standard rotary kilns is possible at a temperature around 1250 °C, and that the coating, liquid phase and proper nodulization can be mastered. The minors i.e. Fe, Al, Mg and SO<sub>3</sub> has to be adjusted based on the quality of the incoming RCP.

The studies on carbonation mechanisms and performance evolution were conducted under conditions simulating industrial settings. These studies revealed a significant extent of carbonation, primarily resulting from the reaction of belite with CO<sub>2</sub>. However, the reaction mechanisms of industrial clinkers depend on appropriate cement design.

The carbonation degree of belite clinker is generally higher compared to Portland-based systems, leading to appreciable changes in mechanical performance. The reaction kinetics of belitic clinker and Portland-based systems are similar during carbonation. However, in the case of Portland cement, hydration reactions continue at a slower pace after carbonation reactions.

The investigation of microstructure evolution indicates discernible differences between the two systems. In both cases, carbonation reactions lead to a decrease in porosity and its refinement, ultimately affecting the mechanical performance formation.

Finally, experiments on cement mortars and concretes revealed that the concrete products based on belite clinkers are characterized by appreciable performance after carbonation curing. The mortars and concretes investigated outperformed the early strength after 1 or 2 days of typical Portland-based systems and bears the potential of good strength at the age of 28 days. Low w/c ratio, optimized density and an adequate curing procedure support this good strength development by carbonation.

All in all, the up-to-date project results demonstrate that the described technology is a promising solution to limit the CO<sub>2</sub> emissions of the cement and concrete industry, with high potential for industrial upscaling.

# Advancing Mineral Carbonation in Cement-based Materials through Tailored Amine Blends

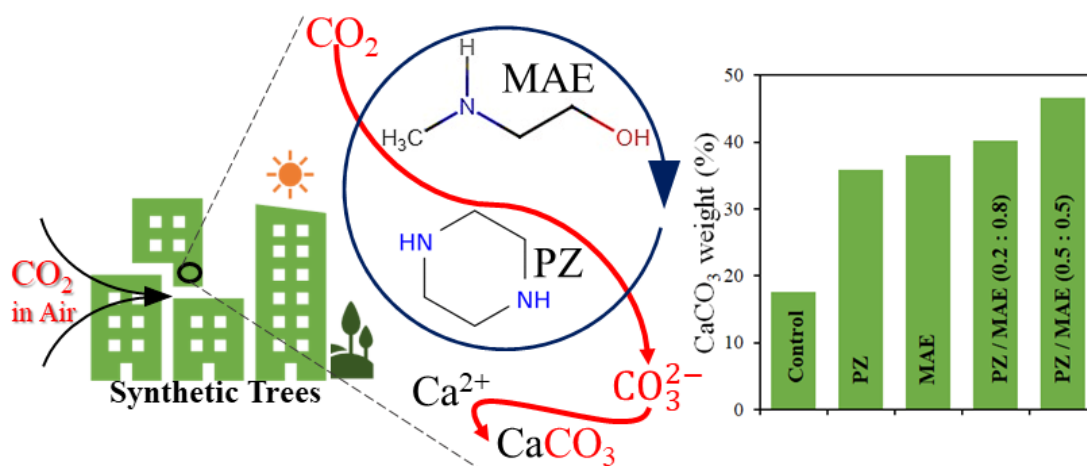
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**Keywords:** Carbon capture; Mineral Carbonation, 2-(methylamino)ethanol (MAE), Piperazine

Mineral carbonation facilitated by aqueous amines is a carbon capture technique applied in global CO<sub>2</sub> emissions controls, featuring enhanced CO<sub>2</sub> dissolution plus mineral carbonate deposition and amine regeneration. As a novel approach, amines blend into cement-based materials can be worked as ‘synthetic trees’ in direct air capture (DAC). Extending this concept, the current work was initially conducted to identify the most potential amine to absorb a high amount of CO<sub>2</sub> using Ca(OH)<sub>2</sub> as the cement-based material. Thermogravimetric Analysis was conducted to analyse the CO<sub>2</sub> capture by CaCO<sub>3</sub> weight %. 2-(methylamino)ethanol (MAE), N-methyldiethanolamine (MDEA), 2-amino-2-methyl-1-propanol (AMP), ethane-1,2-diamine (EDA), and 2-(2-aminoethylamino)ethanol (AEEA) were selected as amines. Solutions with 300 mL of 1 M aqueous amines plus Ca(OH)<sub>2</sub> in 1:10 of solid: liquid ratio were carbonated 2 h at 2.0 L/min of 5 % CO<sub>2</sub> gas flow rate at 20 °C. There, MAE was observed with the highest CO<sub>2</sub> absorption. Yet, considering the chemical structures, blends of amines are expected to deliver further enhancement of CO<sub>2</sub> absorption in the presence of cement-based materials. This study was further focused on the effect of PZ and MAE blends on CO<sub>2</sub> absorption behaviour with Ca(OH)<sub>2</sub>. The PZ/ MAE blends were prepared in different ratios of PZ: MAE as 1.0:0, 0.2:0.8, 0.5:0.5, 0.8:0.2, and 0:1.0 maintaining total amine concentration at 1.0 M. Comparison was made with Ca(OH)<sub>2</sub> without any amine inclusion as the control. As a result, augmented CO<sub>2</sub> capturing for PZ/MAE blends at 0.5:0.5 and 0.2:0.8 PZ: MAE ratios were observed compared to control, as well as to pure PZ and MAE. The highest CaCO<sub>3</sub> weight % was noted for 0.5:0.5, while the second highest was recorded for 0.2:0.8 PZ: MAE ratios. Understanding the reaction mechanism in PZ/ MAE blend with CO<sub>2</sub>, identification and confirmation of CaCO<sub>3</sub> polymorphs and their microscopic structures, and DAC experiments will be conducted to standardise these spotlight findings.



# Effect of carbonation on cementitious materials microstructure by X-ray tomography

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*Keywords: Carbonation; X-ray Tomography; Cement; Recycled aggregate*

The carbonation of recycled concrete aggregates is a crucial approach for sequestering CO<sub>2</sub> emissions generated during production, aiming to enhance circularity and mitigate CO<sub>2</sub> emissions in the cement and concrete industry. Despite its significance, the intricate nature of the carbonation process is influenced by factors such as the transport and reaction of carbon dioxide within the material, both dependent on the moisture content in the pore network. Current theories, relying on point-wise and macroscopic averaged measures, fail to adequately capture the inherently heterogeneous nature of the process, necessitating comprehensive full-field approaches. The impact of factors like relative humidity remains ambiguous, simultaneously reducing CO<sub>2</sub> permeability in pores while enhancing dissolution and carbonation reactions. Additionally, spatial variations in humidity within the material contribute to the complexity.

This study employs two distinct techniques to investigate carbonation processes. Firstly, at the laboratory scale, recycled concrete aggregate samples from construction and demolition wastes are sieved into three size categories (0-200µm, 200-500µm, and 500-4000µm) and subjected to carbonation at 20% CO<sub>2</sub> concentration with varying water content (5%, 10%, and 15%). A comprehensive analysis, including mass gain, TGA, and C/H methods, is conducted to evaluate the quantity of bound CO<sub>2</sub>. This study establishes the correlation between the quantity of bound water and recycled concrete aggregate size as a function of the initial water saturation and reveals a low degree of carbonation in all tested samples.

Secondly, a carbonation chamber is developed to observe the carbonation process operando using X-ray tomography at ID19 (ESRF) and ANATOMIX (SOLEIL) in France. In the proposed experimental campaign, various hydrated cement pastes samples have been exposed to carbonation at 20% and 100% CO<sub>2</sub> concentration, both at 20°C and 80°C. The resulting carbonation front has been tracked under different environmental conditions. Results indicate cracks initiation and propagation due to coupled carbonation-drying shrinkage followed by cracks sealing at the end of the process.

# Assessing the suitability of Fe-rich industrial by-products for the production of carbonate-based binders

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*Keywords: iron-rich, industrial by-products, characterisation*

The carbonation of industrial by-products such as steel slags, has recently become an area of interest for the production of low carbon supplementary cementitious materials and alternative binders. These studies tend to focus on materials rich in calcium (Ca)- or magnesium (Mg)-oxides and silicates, while little research has been dedicated to understanding the carbonation of iron (Fe) and the carbonation potential of widely available Fe-rich resources.

Systems based on or including  $\text{FeCO}_3$  are of growing interest as alternative binders and as many Fe-rich resources also have significant Ca and Mg content, mixed carbonate systems may be realised. Towards this goal Fe-rich materials must be thoroughly characterised to identify which Fe-containing phases are present and under what conditions the phases in the materials could participate in carbonation reactions.

Six steelmaking by-products have been examined in this work, from electric arc furnace (EAF), blast furnace (BF) and basic oxygen steelmaking (BOS) processes. The by-products fall into three categories: steelmaking dusts, slags and filter cakes. XRF, TGA and XRD have been performed on the materials and water content has been established through drying at 200°C until a constant weight is reached. A selection of results obtained to date can be seen in Table 1.

*Table 1: FeO/Fe<sub>2</sub>O<sub>3</sub> content identified through XRF and phases identified through XRD and/or TGA*

Material	Wt% FeO/Fe <sub>2</sub> O <sub>3</sub>	Notable Phases
EAF Red Dust	47	Franklinite, Zincite
EAF Drop out Dust	38	Calcite, Franklinite, Magnetite, Wuestite, Perovskite
EAF Slag	33	Larnite, Brownmillerite, Franklinite, Magnetite
BOS Slag Fines	19	Portlandite, Calcite, Wuestite, Brownmillerite, Magnetite
BF Filter Cake	20	Calcite, Fe, Hematite, Siderite, Wuestite, Zincite, MgSiO <sub>3</sub>
BOS Lagoon Cake	78	Calcite, Fe, Hematite, Siderite, Wuestite, MgSiO <sub>3</sub>

Metallic Fe and Wuestite in the steelmaking cakes indicates good potential for CO<sub>2</sub> uptake by Fe containing phases. Metallic Fe may also provide good nucleation sites for siderite formation and the development of FeCO<sub>3</sub> containing binders. The two slags differ in composition, but both contain phases which should take up CO<sub>2</sub> such as larnite and wuestite. The steelmaking dusts contain less Fe(II) and little Ca so are less promising from a carbonation perspective. However, Wuestite in the drop out dust and zincite in red dust can carbonate. As Zn is a valuable metal, it may be more economically viable to extract Zn from the red dust than use it in carbonation applications.

Dissolution experiments are the next step towards determining the suitability of these materials for carbonation and therefore establishing whether any may be suitable for use as a carbonate binder precursor material.

# Transforming lignite fly ash into a carbon neutral SCM through mineral carbonation

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*Keywords: Lignite Fly Ash, Mineral Carbonation, use of waste materials*

## Context

Cement production generates nearly 9% of global CO<sub>2</sub>, driven by clinker burning. Substituting clinker with mineral additives (SCMs) is key to sustainable cements. Traditionally, fly ash from coal and slag were prominent SCMs, but industry shifts are reducing their availability, necessitating alternatives. Lignite fly ash (LFA), produced in significant quantities (5-10 million tons/year in Germany), emerges as a potential SCM. In addition, much larger quantities have been deposited in landfills, meaning that sufficient material will still be available beyond the planned coal exit in Germany. However, problems may occur due to free CaO, MgO, and varying sulphate contents. These components react in a cementitious environment and form damaging hydroxides.

## Objectives

The ERA-MIN 3 project CO<sub>2</sub>TREAT tackles the previously mentioned challenges associated with the utilization of LFA in cement-based systems. Wet mineral carbonation is used to address the aforementioned challenges. The sample is carbonated under ambient conditions in an aqueous environment with dissolved CO<sub>2</sub>, achieving high reaction yields. Alkaline earth oxides are transformed into carbonates, which permanently bind CO<sub>2</sub> and do not trigger harmful reactions in the cementitious system. In the wet process, sulfate components partially dissolve, which could reduce the formation of secondary ettringite in a cementitious environment.

However, the most significant advantage of mineral carbonation is the carbonation potential of silicate and aluminate-rich phases, such as the amorphous glass phase, akermanite-gehlenite, merwinite, melilite, ettringite, CSH, etc. In the context of carbonation, these phases can form carbonates as well. Additionally, the remaining residues are amorphous aluminate and silicate gels that exhibit pozzolanic reactivity. This allows for the substitution of higher cement contents with fly ash, which could lead to a significantly higher CO<sub>2</sub> reduction potential in a further step.

## Main results

A wet carbonation reactor was built capable of performing carbonation reactions between 30 °C and 70 °C, with a capacity of up to 1.8 L, and CO<sub>2</sub> concentrations ranging from natural conditions to 90 %. A partial factorial parameter study was conducted using this reactor principle to investigate the influencing factors to maximise CO<sub>2</sub> uptake. Phase development was analyzed using in situ investigation of pH, conductivity, carbon-sulfur analysis, X-ray diffraction, and R<sup>3</sup> calorimetry. The results show that 50 to 140 kg CO<sub>2</sub>/tonne of fly ash can be bound. Furthermore, the pozzolanic reactivity is increased by the carbonation reaction and the sulphate content is reduced.

# CO<sub>2</sub> mineralisation of fresh mixture to produce cement and BOF steel slag pastes

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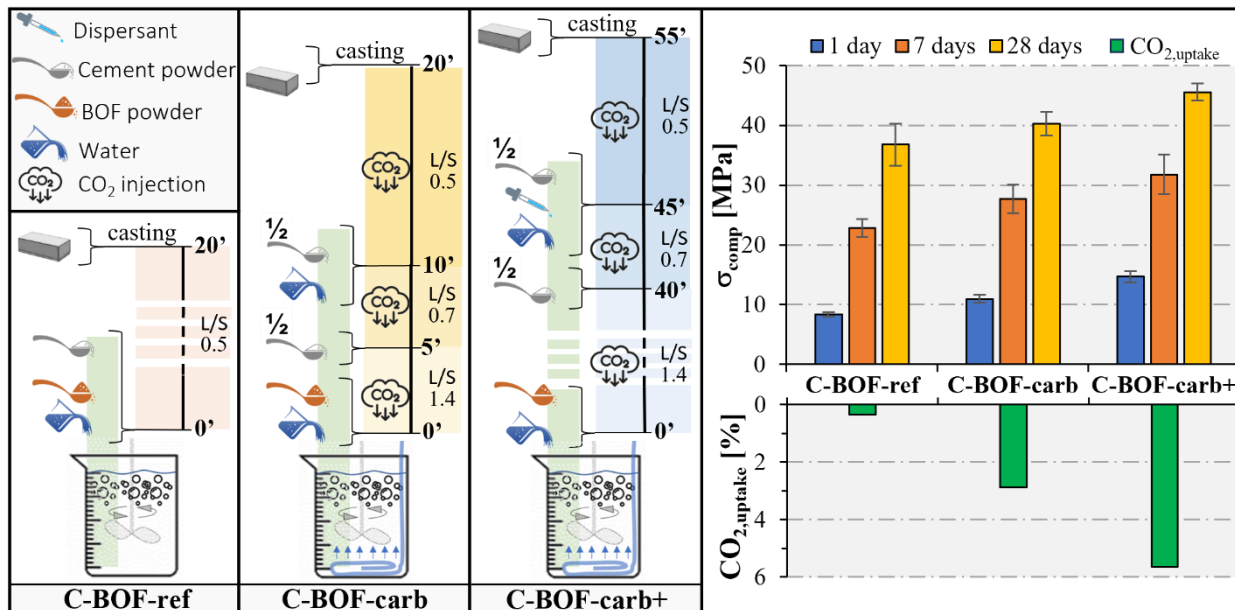
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*Keywords: Carbon Capture and Utilisation; Mineral Carbonation; BOF Steel Slag; Cement carbonation.*

Commonly investigated CCU technologies, produce either carbonated materials to be adopted in fresh mixtures (i.e. powders or aggregates) or carbonated pre-formed manufact (i.e. blocks produced via carbon curing). This study aims at exploring an innovative mineralisation process, still not deeply investigated in the literature, in which the accelerated carbonation is induced by injecting CO<sub>2</sub> gas in fresh mixture to produce pastes.

Cement of type 52.5 N is adopted as binder with 30% in weight substitution of Basic Oxygen Furnace (BOF) steel slag. An apparatus to directly inject the gas during the mixing process is implemented at laboratory scale and adopted to produce mineralised pastes. Carbonated samples, *C-BOF-carb*, are produced with a water-to-binder ratio of 0.5 and by mixing for 20 minutes with a constant CO<sub>2</sub> flow rate of 250 l/h. An improved configuration, *C-BOF-carb+*, was created by pre-carbonating BOF and by using a dispersant additive. The efficiency of the fresh mixture carbonation process is analysed by a comparison with reference samples, *C-BOF-ref*, both in terms of CO<sub>2</sub> uptake and mechanical properties of the hardened products. The experimental outcomes show a CO<sub>2</sub> uptake of the end-product up to 5.7%, and enhanced mechanical properties compared to non-carbonated reference samples. The promising results pave the way for further analysis aimed at improving the process and assessing its efficiency to produce mortar elements.



# Production of cement replacement (SCM) by CO<sub>2</sub>-beneficiation of steel slag

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*Keywords: SCM; steel slag; CO<sub>2</sub> beneficiation; semi-wet carbonation*

Lowering the direct CO<sub>2</sub> emissions of cement production is a prime objective of the cement industry. Substitution of clinker, for instance with by-products from other industries, is a widely adopted means to reduce direct emissions. A bottleneck in lowering clinker content is the availability of known clinker-replacement materials, as beneficiation processes are often required to condition the materials for such use. A novel approach is to use mineral carbonation of steel slags as a beneficiation step for the conversion of the by-product into a supplementary cementitious material (SCM). The reaction with CO<sub>2</sub> activates Ca-silicates by formation of calcium carbonates and reactive silica, immobilizes substances of concern and more importantly, it enables to sequester CO<sub>2</sub> in stable mineral products.

In the ERA-MIN3 project CO<sub>2</sub>TREAT, a new direct (semi-)wet carbonation processes is developed for beneficiation of BOF steel slags. A dynamic treatment process was set up to study the main carbonation mechanisms and identify carbonation rate controlling parameters. The presentation will show the main outcomes of the process, focusing on carbonation degree and reactivity in function of carbonation time, CO<sub>2</sub> pressure, relative humidity and temperature.



# Carbonation of electric arc furnace steel slag under hydrothermal condition

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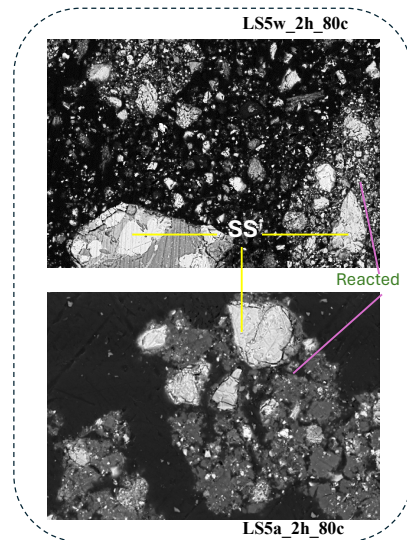
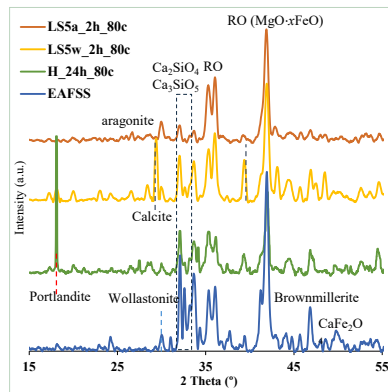
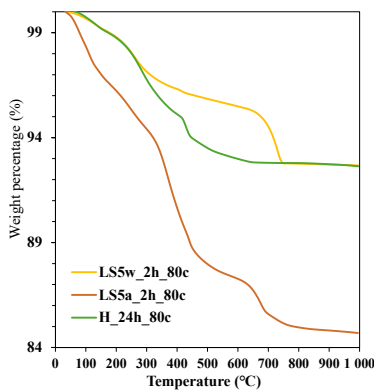
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*Keywords: Steel slag; Hydrothermal reaction; Carbonation; Supplementary cementitious materials*

Using steel slags as supplementary cementitious materials presents a promising opportunity to substantially reduce CO<sub>2</sub> emissions in the cement industry. Carbonation treatment of steel slag not only can activate its phases involving in early hydration but also has the function of carbon sequestration. This study tried to improve the carbonation efficiency of steel slag by different hydrothermal treatment methods. The chemical composition of mineral of electrical arc furnace steel slag (EAFSS) is presented in the figure. The EAF slag was put in a sealed reactor with a liquid to solid ratio of 5, and then the treatment was operated under three different hydrothermal conditions, including 24 hours at 80 °C (H\_24h\_80c), hydrothermal reaction under same temperature at 5 bar CO<sub>2</sub> pressure for 2 hours (LS5w\_2h\_80c) and the same condition in a solution of 2 mol/L acetic acid (LS5a\_2h\_80c). The composition of treated EAFSS was measured by Thermogravimetric analysis, X-ray diffraction and Scanning electron microscopy.

Chemical composition of EAF slag

Chemical composition	Fe <sub>2</sub> O <sub>3</sub>	CaO	SiO <sub>2</sub>	MgO	MnO	Al <sub>2</sub> O <sub>3</sub>	Cr <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	SO <sub>3</sub>	P <sub>2</sub> O <sub>5</sub>	V <sub>2</sub> O <sub>5</sub>	Nb <sub>2</sub> O <sub>5</sub>
Weight percent	37.70%	29.10%	9.71%	6.32%	6.20%	4.92%	2.24%	0.40%	0.35%	0.30%	0.20%	0.12%



Results show that the hydrothermal reaction at 80 °C largely dissolved calcium from silicates, which leads to precipitation of portlandite. However, the RO (MgO-xFeO solid solution) phase is rarely dissolved even after 24 hours reaction. The hydrothermal reaction under the 5 bar CO<sub>2</sub> condition results in the formation of calcite. The addition of acetic acid appears to increase the dissolution of calcium silicates while inhibiting the precipitation of divalent metal carbonates, as indicated by the reduced mass loss between 600 °C to 800 °C in thermogravimetric analysis. Scanning electron microscopy images show a notable enhancement in the reaction degree of steel slag (SS) in the presence of acetic acid. The large mass loss between 300 °C to 500 °C seems to result from the decomposition of calcium and magnesium acetate. However, there is no corresponding peak detected in XRD for these two salts. Therefore, further investigation is needed to clarify the composition of hydrothermally treated steel slag under CO<sub>2</sub> atmosphere. Additional conditions will be explored to enhance the decomposition of the RO phase, aiming to activate these phases for use as supplementary cementitious materials.

# Advancing Carbonation Tracking: The Pivotal Role of Carbon Isotope Analysis in Modern Techniques

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**Keywords:** Wet-carbonation of cementitious phases, carbon isotopy, calcite, vaterite,  $\delta^{13}\text{C}$

This study investigates the wet carbonation of  $\text{C}_2\text{S}$  in a closed system at room temperature, employing techniques such as quantitative X-ray diffraction (QXRD) and thermogravimetric analysis (TGA) to monitor the carbonation process. It also involved measuring pH, calcium and silicon concentrations, modeling carbon species in solutions, and determining the saturation indices of phases.

The new innovative approach in the study to understanding the carbonation reaction comes from the measured stable carbon isotopes in the system. We measured  $\delta^{13}\text{C}$  in  $\text{CO}_2$ , dissolved inorganic carbon (DIC), and carbonate phases to explore the potential fractionation of  $\delta^{13}\text{C}$  isotopes during carbonation and whether this fractionation could be linked directly to the carbonation reaction. The findings indicate that  $\delta^{13}\text{C}$  isotopes undergo fractionation throughout the carbonation process, which spans 48 hours in our experiment, transitioning from the gaseous phase to the solution and finally to the solid phase, with isotopic fractionation correlating with the carbonation's progress (see figure 1).

The research highlighted the formation of three carbonation products within 48 hours: calcium carbonate (in calcite and vaterite forms) and amorphous silica gel. The carbonation process was divided into three stages measured through  $\text{CO}_2$  consumption and the phase content of  $\text{C}_2\text{S}$  and  $\text{CaCO}_3$ , revealing detailed insights into the reaction dynamics and the potential of  $\delta^{13}\text{C}$  isotopes for tracing carbonation pathways and reaction timelines.

Future studies delved into the distinctiveness of  $\delta^{13}\text{C}$  fractionation during carbonation and its ability to differentiate between  $\text{CaCO}_3$  sources, which is crucial for accurately determining  $\text{CO}_2$  uptake in carbonation, particularly in materials that already contain  $\text{CaCO}_3$  as aggregate before carbonation. This research opens new avenues for understanding and quantifying carbonation processes, with implications for carbon capture and storage technologies.

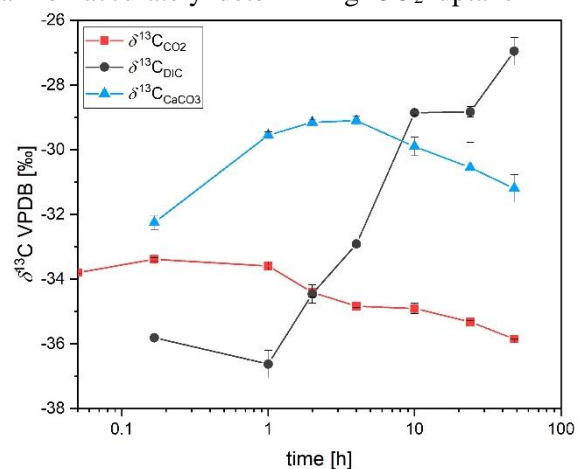


Figure 1:  $\delta^{13}\text{C}$  fractionation during wet carbonation of  $\text{C}_2\text{S}$

# Carbonation behaviour of recycled fines derived from blended cementitious materials: composition, microstructure and mechanism

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*Keywords: recycled concrete fines; accelerated carbonation; supplementary cementitious materials*

Waste concrete has been recycled and crushed to produce recycled concrete fines (RCFs) as sustainable alternatives to natural sand. Accelerated carbonation has been integrated with the recycling of RCFs to improve their qualities and to sequestrate CO<sub>2</sub>. However, RCFs derived from plain concrete were the main focus of previous investigations. Precise ideas about the carbonation behaviours of RCFs originating from blended concrete with supplementary cementitious materials (SCMs) have been scarce.

Therefore, in this study, five types of simulated RCFs made from blended cement paste were prepared based on three traditional SCMs at varying ratios. The RCFs were carbonated and their differences in carbonation efficiency, microstructure, mineralogy, and physical properties were examined. The results demonstrated that the behaviours of RCFs towards carbonation were both SCM- and dosage-dependent, and they were intrinsically associated with the initial amount of portlandite, the Ca/Si ratios of C-S-H, the presence of aluminate hydrates, as well as the compactness of initial microstructures. Specifically, RCFs derived from fly ash (FA) and silica fume (SF) blended cement paste have higher carbonation rate of up to 62.4% than plain RCFs. Despite such an improvement, increasing FA dosages (i.e., 30%) caused decreasing benefits and even adverse impacts on RCFs microstructural densification. This result was attributed to the silica- and alumina-rich nature of FA that depleted portlandite, causing the predominant decalcification on low Ca/Si C-S-H and Aft/AFm during carbonation which were more likely to cause solid volume decrease. Generally, the enhanced access of carbonate species to the inner RCFs matrices due to the deterioration of microstructure during carbonation was considered as both the consequence and driving force for the poor behaviour of RCFs containing FA. By contrast, RCFs derived from ground granulated blast furnace slag (GS) blended cement paste exhibited an opposite trend i.e., lower carbonation rate and degree as compared to plain RCFs. It was critically owing to the compact initial microstructure of these RCFs, thus possessing a high resistance to carbonate species. Carbonation on these RCFs induced limited change in both mineralogy and microstructure. In terms of surface morphologies, vaterite formation was favoured in the presence of FA, the size of calcite grains was restrained by SF, and vesuvianite formed when GS/FA that contained a rich amount of alumina was present. Although mineralogical and microstructural changes were different, the water absorption of all RCFs were found to reduce, possibly due to the densification of microstructures or the formation of the dense Cc deposition rim.

Overall, the results presented in this study suggested that the carbonation behaviours of RCFs from different origins had dramatic differences. The improvement of carbonated RCFs as compared to raw RCFs became less significant and even slightly adverse due to the presence of some SCMs in the parent materials. As such, the understanding of this study could provide a foundation for the strategic adjustment and systematic development of carbonation treatment for RCFs.

# Upcycling different sources of RCP into a carbon negative SCM through mineral carbonation

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*Keywords: SCM; recycled concrete powders (RCP); CO<sub>2</sub> beneficiation; semi-wet carbonation*

Substitution of clinker by supplementary cementitious materials (SCM) valorized from other industries' by-products has been widely used to lower the CO<sub>2</sub> emission of cement production. Many of the conventional SCM's such as silicious and calcium rich fly ashes and ground granulated blast-furnace slag (GGBFS) are fully in demand. However, due to energy transition, the industries producing these SCM will close or will undergo technological changes to become less energy intensive.

It's important for the construction industry to stay adaptable and responsive to these potential changes, by investing in research and development to discover new SCM's, mitigating potential SCM shortages. A novel approach that has been investigated in the last several years is the use of mineral carbonation as a beneficiation step for the conversion of by-products, into SCM's. The reaction with CO<sub>2</sub> activates Ca-silicates by formation of calcium carbonates and reactive silica. Significant work has been carried out to investigate potential reactivity of recycled concrete fines (RCP) mostly using the wet carbonation technique. In this study the effect of cement type on the carbonation of RCP to produce SCMs is investigated. Static and dynamic carbonation are used to investigate RCP, originally produced with a CEM I 52.5 and CEM III 52.5 N. In this paper, the results of an experimental campaign to better understand the effect of process conditions for carbonating the RCP, with the aim of obtaining the highest degree of carbonation and reactivity, are presented.

# Exploring the Potential of Mineral Carbonation in Cement Production with Carbonated SCMs

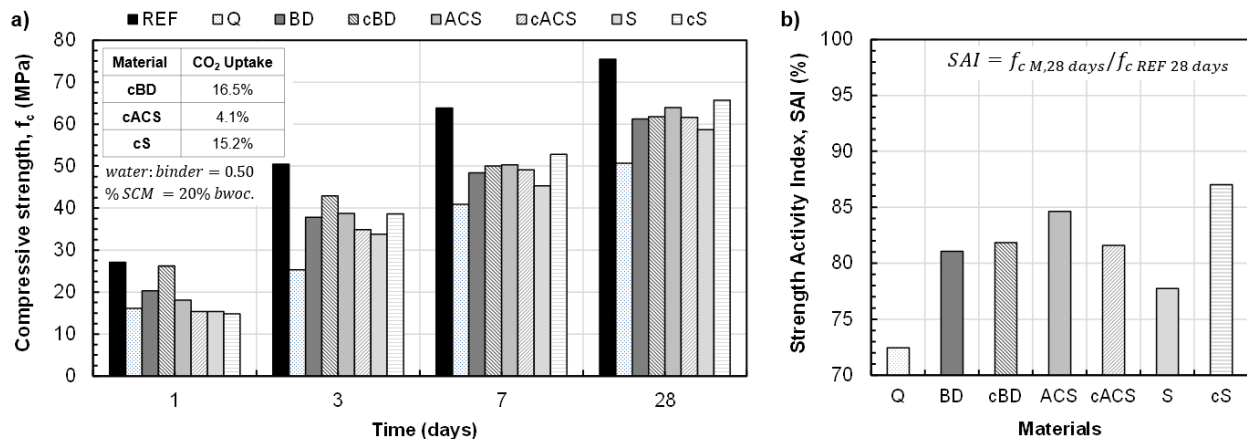
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**Keywords:** Cement; Mineral Carbonation; Supplementary Cementitious Materials.

In this article, we explore the potential of mineral carbonation in enhancing the sustainability of cement production. Mineral carbonation, a carbon sequestration technology, repurposes CO<sub>2</sub> emissions into stable carbonates and has significant potential in the reclamation of industrial byproducts such as bypass dust and steel slags. Our research investigates the process of transforming these byproducts into carbonated Supplementary Cementitious Materials (cSCM). We present an experimental investigation of carbon uptake from bypass dust (BD), basic oxygen steel slag (S), and air-cooled steel slag (ACS) and examine the mechanical performance of blended cement mixed with SCM and cSCM at a 20% mass replacement.

The tested materials are mixed with water at a 1:25 mass ratio (solid : water) and carbonated for 60 min in a 40L stirred media reactor equipped with a steel-rod impeller (1600 RPM) and injection inlet that pumps a simulated flue gas – 20% CO<sub>2</sub> + 80% Air (ca. 0.8 N<sub>2</sub>+0.2 O<sub>2</sub>) – at 60 L/min. The reaction time is set at 60 min, coinciding with the slurry solution's pH stabilization. The CO<sub>2</sub> content in the fresh and carbonated materials is measured by a Calcimeter and converted into CO<sub>2</sub> uptake (i.e. CO<sub>2</sub> mass / dry material mass).



**Fig. 1.** Test results: a) compressive strength ( $f_c$ ) and CO<sub>2</sub> uptake and b) SAI for  $f_{c, 28 \text{ days}}$ .

Fig. 1 depicts the compressive strength tests and CO<sub>2</sub> uptake and for mortars prepared with materials before and after carbonation. The mix REF is the baseline mortar with 100% CEM I 52.5N, while Q is a mortar where 20% quartz ( $\varnothing_{\text{max}} < 90\mu\text{m}$ ) replaces cement – where quartz is accounted for as an inert material.

Fig. 1 data shows that both fresh and carbonated byproducts contribute to  $f_c$  increase from 7 days on for the tested replacement ratio, since the  $f_c$  values for the SCM and cSCM mixes are greater than that from the inert mix Q. In relation to the REF mix, all byproducts classify as pozzolanic, since their Strength Activity Index (SAI, in Fig 1b) is greater than 75% at 28 days (ASTM C618). On the impact of mineral carbonation of SCMs, our results show that it helps improve SCM reactivity – except for ACS, where the SAI is reduced from 85 to 82% after carbonation. S provided the best overall result, where the SAI<sub>cS</sub> increase to 87%.

Altogether, our research provides valuable data regarding CO<sub>2</sub> sequestration rates in industrial byproducts as well as insights on the valorisation of these as a cSCM, which could support the production of a more sustainable cement where industrial byproducts are available.

# Effect of triethanolamine on carbonation of clinker-free Class C fly ash

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*Keywords: Fly ash; CO<sub>2</sub> curing; Triethanolamine; Compressive strength*

This study investigated the effect of triethanolamine on the carbonation and hydration of Class C fly ash. As the international agreement against climate change proposed that the world must achieve net-zero CO<sub>2</sub> emissions by 2050, the development of technology to reduce CO<sub>2</sub> is necessary. Fly ash, a byproduct from coal fire plants, has been used as supplementary cementitious materials (SCMs) to lower clinker, however, the clinker production should be further reduced and the technology on carbon capture, utilization, and storage should be introduced in construction materials area to achieve carbon neutrality. Because fly ash itself can gain compressive strength without clinker involvement through carbonation [1], it was considered a green construction material.

Since carbonation is affected by the calcium content and TEA can enhance the calcium solubility of fly ash, the addition of TEA led to a noticeable increase in the compressive strength of carbonated fly ash pastes. For instance, in the addition of TEA 5 wt.%, compressive strength was improved up to 70 MPa on 7 days of carbonation (Note Figure 1). Meanwhile, TEA could also improve the compressive strength of moisture-cured fly ash pastes, but not as much as carbonated fly ash at the same amount of TEA addition. In the presence of TEA, ettringite and AFm phases were formed in moisture curing, while C(-A)-S-H and amorphous calcium carbonate were produced in carbon curing, which led to the refinement of porosity in both curing conditions.

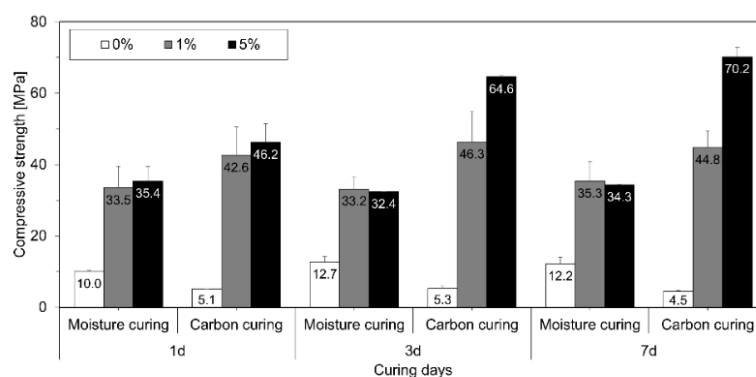


Figure 1. Compressive strength of fly ash pastes cured in carbonation and moisture conditions at 0, 1, 5% TEA addition.

## Reference

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# Use of carbonated recycled aggregate and concrete slurry waste in recycling concrete – results of the Swiss "DemoUpCarma"-project

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## Abstract

Within the framework of the "DemoUpCarma"-project (<http://www.demoupcarma.ethz.ch>), a pilot project to study CO<sub>2</sub> management solution for a net-zero Switzerland, the domestic storage of CO<sub>2</sub> in recycled concrete was investigated. In laboratory experiments, recycled concrete aggregates (RCA) and concrete waste slurry were carbonated using 100% CO<sub>2</sub>. In parallel, real-scale installations to carbonate both materials were implemented at a local concrete plant. In case of the RCA, different moisture saturation levels were considered, which is relevant for the real situation at the concrete plant. The carbonated materials were used to produce recycling concrete with a lower CO<sub>2</sub>-footprint than conventional recycling concrete.

The main findings can be summarized as follows:

About 10-13 kg CO<sub>2</sub> per t of dry RCA (0-16 mm) can be absorbed at moisture contents of practical relevance (60-200 % of the aggregate's water absorption). Smaller size fractions (i.e. the 0-4 mm fraction) are able to absorb significantly more CO<sub>2</sub> than larger fractions. The carbonation leads to a patchy distribution of decalcified C-S-H on the surface of the RCA particles, which can participate in cement hydration. Concretes with carbonated RCA loose workability more rapid than those with non-carbonated RCA, but yield a higher compressive strength.

Carbonated concrete waste slurry can adsorb 120-130 kg CO<sub>2</sub> per t of dried material. It shows a rapid early pozzolanic reaction.

As both carbonated RCA and concrete waste slurry increase concrete performance, the cement content in the recycled concrete can be lowered. This provides an additional reduction of the CO<sub>2</sub> footprint besides the one due to the stored CO<sub>2</sub>, when compared to conventional recycling concrete.

## Keywords

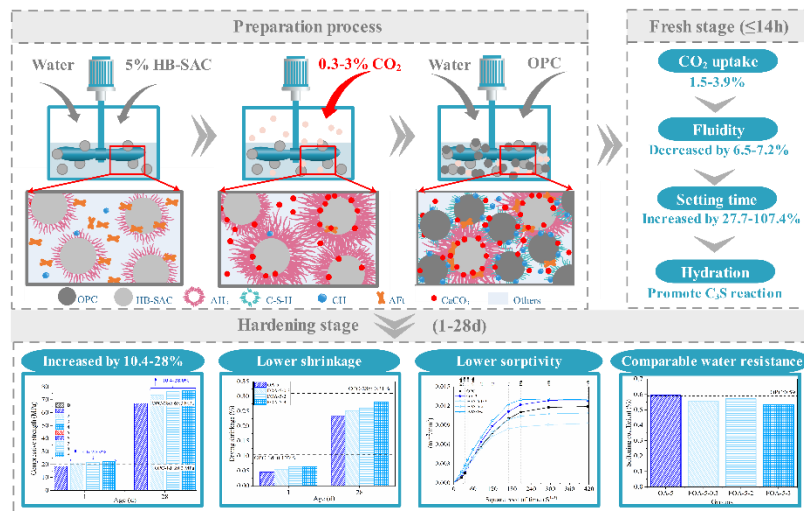
CO<sub>2</sub>-reduction, recycling concrete, carbonated recycled aggregates, carbonated concrete waste slurry

# Improving the Mechanical Properties of Portland Cement via Blended CO<sub>2</sub>-intermixed High Belite Calcium Sulfoaluminate Cement

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*Keywords: CO<sub>2</sub> intermixing, high belite calcium sulfoaluminate cement, compressive strength, reaction mechanism*



Intermixing CO<sub>2</sub> during the mixing process (CO<sub>2</sub>-intermixed) of ordinary Portland cement (OPC) is a feasible way to enhance early mechanical properties. However, the slow reaction between CO<sub>2</sub> and tricalcium silicate, coupled with excessive carbonation products (CaCO<sub>3</sub>) on the cement clinker's surface, reduces the CO<sub>2</sub> uptake and long-term strength of OPC paste. This study introduces a novel CO<sub>2</sub> intermixing approach utilizing high belite sulfoaluminate cement (HB-SAC), known for its rapid hydration rate and CO<sub>2</sub> reactivity, to enhance the mixture's properties. Initially, 5 % HB-SAC was mixed with water and then intermixed with 0.3-3% CO<sub>2</sub>. Subsequently, OPC and the remaining water were added to prepare the blended mixtures (FOA). For comparison, non-carbonated HB-SAC blended OPC paste (OA) with W/B 0.4 were also prepared. The results show that the present CO<sub>2</sub> in the HB-SAC paste promotes the generation of aluminum hydroxide (AH<sub>3</sub>) gel and ettringite, causing decreased fluidity in the FOA paste. In contrast to the OA group, CO<sub>2</sub> uptake in the FOA group increases by 53.1% at a 3% CO<sub>2</sub> dose. Notably, higher CO<sub>2</sub> doses significantly enhance the exothermic reaction of C<sub>3</sub>S, resulting in a higher C<sub>3</sub>S peak compared to C<sub>3</sub>A. Consequently, after one day of curing, the FOA group intermixed with 3% CO<sub>2</sub> exhibits a 28% higher compressive strength than the OA group. Over the subsequent 28 days, FOA groups exhibited a higher content of calcium silicate hydrate (C-S-H) and AH<sub>3</sub> gel compared to OA groups due to accelerated hydration of calcium silicate phases (C<sub>2</sub>S and C<sub>3</sub>S) and calcium sulfoaluminate (C<sub>4</sub>A<sub>3</sub>S), effectively densifying the blended cement paste's microstructure. As a result, the sorptivity and water resistance of FOA paste improve. In summary, blending 5% CO<sub>2</sub>-intermixed HB-SAC with 95% OPC effectively enhances hydration and carbonation reactions, leading to superior comprehensive properties.



# Growth of calcium carbonate in recycled cement paste induced by different accelerated carbonation regimes: the role of water

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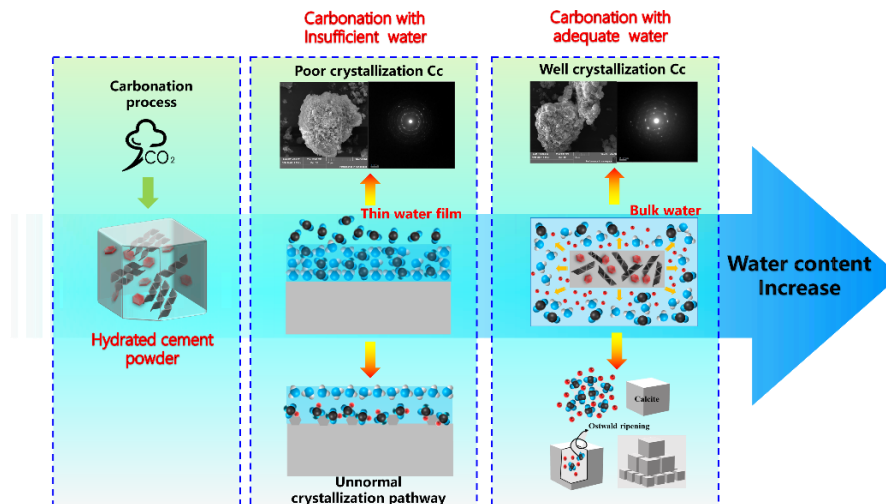
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**Keywords:** Recycled cement powder; Carbonation; polymorphs; Calcium carbonate; Crystallization

High-alkaline waste cement possesses significant potential for effectively capturing and sequestering carbon dioxide, transforming it into calcium-rich, highly reactive supplementary cementitious materials. Water plays a pivotal role in facilitating this reaction, yet there is a conspicuous absence of extensive research in this domain. In this study, the water content during carbonation gradually increased from dry carbonation at 55% relative humidity to wet carbonation with a liquid-solid ratio of 40. The study elucidates the mechanisms by which water affects the growth behavior (precipitation and crystallization) of calcium carbonate (Cc) in hydrated cement paste during carbonation.

The findings indicated that the presence of water dramatically affected Cc's polymorphs, morphology, and crystal properties. With an increase in water content from RH=55% to a liquid-solid ratio of 40, the calcite grain size underwent significant enlargement from less than 200nm to over 400nm. The simultaneous decalcification of CH and C-S-H was observed in dry carbonated cement paste, whereas the decalcification of C-S-H commenced after the depletion of CH in wet carbonation. Moreover, as the water content increased, the different Cc polymorphs evolved into mainly calcite, and the polycrystalline Cc was transformed into monocrystalline calcite. Insufficient water elevated the local pH and ion concentration, resulting in prolonged supersaturation duration. Meanwhile, nano-sized water film when water was insufficient restricted the Cc growth and hindered Ostwald ripening, leading to the preservation of amorphous calcium carbonate (ACC) and Cc with poor crystallization. Overall, the study demonstrated that the crystallization pathways are distinct in different water content environments during carbonation. Insufficient water hinders the growth of Cc, while high water content ensures the dissolution and recrystallization processes to facilitate Cc growth.



# Carbon uptake potential and reactivity of synthetic calcium magnesium aluminosilicate glasses

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*Keywords: CCU, pozzolanic reaction, SCMs, synthetic glasses*

In order to achieve zero-carbon concrete, high replacement levels of cement by SCMs as well as carbon capture and utilization (CCU) methods need to be exploited as much as possible. Enforced carbonation of calcium aluminosilicate glasses has the ability to shed light on the complex carbon uptake behavior in supplementary cementitious materials (SCMs). In addition to the carbon uptake, the process could lead to an increase in reactivity of SCMs through the formation of limestone and a calcium-poor Si-Al-gel. In this study, we use synthesized calcium aluminosilicate glasses with a range of compositions and investigated their carbon uptake potential and the effect of enforced carbonation on their reactivity.

For this, various compositions of synthesized glass powders (the compositions of the glass powders cover a range of Ca/Si and Al/Si ratios) were carbonated using different methods ranging from moist carbonation to wet carbonation to autoclaving. The effectiveness of the processes was evaluated by comparing the carbonated and uncarbonated glass powders using thermogravimetric analysis and several other methods. The reactivity of the powders before and after carbonation is investigated by applying the modified R<sup>3</sup> test.

Results showed that CO<sub>2</sub> uptake in calcium aluminosilicate glasses was negligible. This is an interesting finding as SCMs are commonly studied for CO<sub>2</sub> uptake, and our results show up to 25% CO<sub>2</sub> uptake in SCMs. The reasons that the synthetic glasses do not show uptake are being investigated. It is possible that minor impurities drive the uptake, or that crystalline and amorphous phases show very different uptake behaviors. The results will provide a better insight into the type of materials that are most promising in terms of CO<sub>2</sub> uptake and pozzolanic reactivity to be applied as SCMs after a CCU treatment. These findings have important implications in the emergence of SCMs contributing to zero-carbon concrete in the future.

# Quantification and stability of hydrous carbonate sites in calcite formed by aqueous carbonation of cement pastes

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Aqueous carbonation of cement pastes has been receiving growing attention as a CO<sub>2</sub> mineralization method since it allows CO<sub>2</sub> sequestration from point sources and produces a composite material of calcium carbonates and an alumina-silica phase with pozzolanic properties. Recent studies have mostly focused on carbonation conditions and applications of this method for production of new types of SCMs. However, less attention has been paid to the carbonate phases, which generally are dominated by calcite, the most thermodynamically stable CaCO<sub>3</sub> polymorph. In this study, the formation, stability, and quantification of calcium carbonates formed in carbonated blended cement pastes are investigated primarily by solid-state <sup>13</sup>C NMR.

The formation of carbonates is very rapid and occurs spontaneously as a result of the fast dissolution of Ca-bearing phases and wide availability of CO<sub>3</sub><sup>2-</sup> ions in aqueous carbonation experiments. Multiple techniques indicate the formation of calcite as the main form of calcium carbonate. Evidences from IR spectroscopy, DSC and combined TG/XRD analyses point towards the absence of amorphous calcium carbonates (ACC) in significant amounts. In this study, a hydrous carbonate environment is identified by <sup>13</sup>C{<sup>1</sup>H} CP/MAS NMR experiments and is assigned to carbonate sites at the surface of calcite, close to chemically bound water molecules. This chemical environment is formed mainly during the very early carbonation process, which suggest that its formation is associated with the supersaturated condition of the solution. The hydrous carbonate sites are also stable, not converting to other sites during carbonation, storage and reactivity test conditions.

Quantification of the calcium carbonate phases is carried out by combined <sup>13</sup>C and <sup>13</sup>C{<sup>1</sup>H} CP/MAS NMR experiments and indicates that the hydrous carbonate sites at the surface of calcite corresponds to 18 to 33% of the total carbonate content in carbonated pastes. Carbonation time, chemical composition of the original paste and temperature are seen to influence this proportion.

*Keywords: Calcite; CO<sub>2</sub> sequestration; solid-state NMR; supplementary cementitious materials.*

# Quantification of inorganic carbon in cementitious materials: A report of Japanese project

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*Keywords: Thermal Analysis; Carbonation; Calcium carbonate*

Carbon neutralization in the concrete sector is essential for concrete to be a leading construction material in the construction market. It has been proposed to fix CO<sub>2</sub> in concrete as inorganic carbonates and consider it an offset against CO<sub>2</sub> emissions. In considering such a market, methods to quantify the amount of inorganic carbonate fixation in cementitious materials and its standardization play an important role in this market. In this study, we apply different methods to different concrete component materials, compare the results with each other, and discuss their applicability. Based on the limitation of the applicability of the carbonated hardened cement paste, a new quantification method for carbonated cement paste is proposed.

Table Possible errors found in the combination of method and material.

Method	Aggregate		Binder			Carbonated cement paste
	Sandstone	Limestone	OPC	Fly ash	Blast furnace slag	
Back titration	Calcium carbonate that exists inside the particle may underestimate the results. A finer particle is better.	-	-	Carbon existing inside the particle may influence the results. Crushing of particles is required.	Sulfur may affect the result. Appropriate filtering is necessary.	Quantitative values may include CO <sub>2</sub> adsorbed on silica gel, which is produced during the dissolving process. If the particle size is large, silica-gel may encapsulate the carbonate.
TGA	Clay mineral decomposition may overlap calcium carbonate decomposition.	-	-	Unburned carbon that exists inside particles may overestimate the results.	Oxidation of sulfur may underestimate the results.	The temperature of calcination is unclear, which may over or underestimate the result.
T-C	Calcium carbonate that exists inside the particle may underestimate the result.	Organic and elemental carbon that exist inside particles may overestimate the results.	-	Unburned carbon that exists inside particles may overestimate the result. Sulfur may overestimate the quantification by IR sensor.	Sulfur may overestimate the quantification by IR sensor.	Decarboxylation at low temperatures from ACC / some calcium carbonate is not included in TIC900, which underestimates the result.
TOC	Calcium carbonate that exists inside the particle may underestimate the result. A finer particle is better.	Organic and elemental carbon that exist inside particles may overestimate the result.	-	Unburned carbon that exists in inside particles may overestimate the result. Sulfur may overestimate the quantification by IR sensor.	Sulfur may overestimate the quantification by IR sensor.	Decarboxylation at low temperatures from ACC / some calcium carbonate is not included in TIC900, which underestimates the result.

# Carbonated Construction Demolition Material based on concrete sludge as new Supplementary Cementitious Material in cement

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*Keywords: Carbonation; Cement; Supplementary cementitious material*

Mineral carbonation is identified as a promising technology to reduce carbon footprint of building materials. In this study, Construction Demolition Material (CDM) consisting in pressed dewatered concrete sludge waste was used as raw material. This raw material is then carbonated following two different processes: the first at lab scale using gas with 100% CO<sub>2</sub> and controlled temperature / humidity conditions and the second at pilot scale. This pilot facility uses direct flue gas coming from cement plant (exhaust gas coming from chimney) for carbonation process. Whatever the operating methods, CO<sub>2</sub> reacts with CDM and, calcium carbonate is formed that provides a permanent CO<sub>2</sub> storage in this waste product. The amount of CO<sub>2</sub> sequestrated is related to the cement paste content in CDM and is significant: around 80 to 100 kg CO<sub>2</sub> / t. of initial CDM. This carbonated product was then used as Supplementary Cementitious Material (SCM) at a substitution level of 25% by mass of pure CEM I. Contrary to non-carbonated sample that behaves as limestone filler regarding its reactivity in cement, a higher reactivity of the carbonated sample is measured with a pozzolanic / activity index close to fly ash one . A subsequent thermal treatment at medium range around 450°C of the carbonated sample enables to reach even higher activity index between fly ash and Granulated Ground Blast Furnace Slag. These promising results highlight that carbonated CDM followed or not by thermal activation can be used as alternative SCM in cement without compromise in mechanical performance. Carbonation processes enable the valorization of recycled CDM as SCM in cement and with additional contribution to the reduction of carbon footprint of the binder by permanent CO<sub>2</sub> storage.

# Determination of the CO<sub>2</sub> Uptake of Construction Products Manufactured by Mineral Carbonation

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*Keywords: mineral carbonation; CO<sub>2</sub> uptake; construction materials*

Mineral carbonation is a technology for capturing and storing CO<sub>2</sub> in solid minerals. When mineral carbonation is used to produce construction materials, the quantification of the CO<sub>2</sub> uptake of these products is of the utmost importance, as it is used to calculate the CO<sub>2</sub> footprint of the product and/or carbon offset. The CO<sub>2</sub> uptake is generally determined by measuring the CO<sub>2</sub> content of a material before and after accelerated carbonation. This approach, however, does not take hydration and dehydroxylation reactions into account that may occur during carbonation, and it can therefore under- or overestimate the CO<sub>2</sub> uptake. Thus, a more accurate and practical method to determine CO<sub>2</sub> uptake, which also accounts for hydration and dehydroxylation reactions, is proposed in this paper.

Calculations based on the carbonation of pure minerals showed that not taking dehydroxylation and hydration reactions during carbonation into account may over- or underestimate the CO<sub>2</sub> uptake by up to 32.3% and 43.4%, respectively. Construction materials or waste products rich in Ca(OH)<sub>2</sub> or Mg<sub>2</sub>SiO<sub>4</sub> are most prone to over- or underestimation of CO<sub>2</sub> uptake.

To calculate the CO<sub>2</sub> uptake more accurately, the equation should also take into account the mass change of the initial solid product that occurs during carbonation. Because processes other than CO<sub>2</sub> uptake may lead to weight changes during the carbonation process (e.g., crystal water can be released as free water during carbonation, or free water may become part of the crystal structure), the weight of the products before and after carbonation should be established on a dry weight basis. The conversion of CO<sub>2</sub> content to CO<sub>2</sub> uptake (in g/100 g dry, initial material) is given by Equation:

$$\text{CO}_2 \text{ uptake (wt. \%)} = \text{CO}_{2 \text{ carb.}} \text{ (wt. \%)} \times \frac{\text{weight after carb. (g)}}{\text{weight before carb. (g)}} - \text{CO}_{2 \text{ init.}} \text{ (wt. \%)}$$

with CO<sub>2 carb.</sub> being the CO<sub>2</sub> content in g/100 g of dried carbonated material, and with CO<sub>2 init.</sub> being the CO<sub>2</sub> content in g/100 g of dried initial material, i.e., before carbonation. The “weight before carbonation” is the dry weight of the initial material, and the “weight after carbonation” is the dry weight of the material after carbonation.

Taking into account the policy incentives to encourage and monetarize CO<sub>2</sub> storage through mineralization, it is of utmost importance to determine CO<sub>2</sub> uptake during mineral carbonation in a correct way.

# Progress of NEDO Moonshot Project “Calcium Carbonate Circulation System in Construction”

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Masaki Tamura<sup>4</sup>, Satoshi Fujimoto<sup>5</sup>, Masto Tsujino<sup>6</sup>, Haruo Nakazawa<sup>6</sup>, Yasuhiro Kuroda<sup>6</sup>,  
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*Keywords: Calcium carbonate; Precipitation; Concrete waste; CO<sub>2</sub>; Resource circulation*

Concrete is an indispensable construction material, but its production uses a large amount of limestone and emits a large amount of CO<sub>2</sub>. To fundamentally solve the problems, Ca in concrete is regarded as a potential unused resource capable of capturing CO<sub>2</sub>. By developing a technology to regenerate waste concrete and CO<sub>2</sub> in the air as calcium carbonate concrete (CCC), a new resource recycling system called “C<sup>4</sup>S”, Calcium Carbonate Circulation System for Construction will be realized. This poster outlines a status of the project. Efficient methods for crushing waste concrete and capturing CO<sub>2</sub> are being developed. Crushed waste concrete is separated into particles of a certain size range with no distribution and is then repeatedly exposed to moisture supply and drying in the atmosphere to accelerate the carbonation. The carbonated particles are mixed with a calcium bicarbonate solution produced by blowing an atmospheric CO<sub>2</sub> into water in which carbonated particles have been placed. The mixture is then packed densely into a 10 cm diameter cylindrical mold under pressure to harden to some extent, followed by drying in air and curing in the calcium bicarbonate solution to complete the hardened body of CCC with 20 MPa compressive strength. The cylindrical CCCs are connected in layers and several of them are bundled together and then prestressed to form a column member. Currently, a rigid-frame structure consisting of columns and beams and a wall structure is being considered for buildings using CCC, and an exhibition structure is scheduled to be constructed in 2025. In addition, various studies are being conducted to implement C<sup>4</sup>S in society in 2030, including optimum recycling scenario and analysis of LCCO<sub>2</sub> reduction effect. With the realization of C<sup>4</sup>S, concrete could be permanently utilized in the construction and global warming would be greatly suppressed in the future.

# Investigation of Sample Preparation and Quantification of CO<sub>2</sub> Sequestered Recycled Concrete Aggregates

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*Keywords: Accelerated carbonation; CO<sub>2</sub> sequestration; Carbonated Recycled Aggregates; Thermal analysis, Sample preparation*

The research project “CO<sub>2</sub>crete”, with financial contributions by the Swedish Agency for Economic and Regional Growth and the European Union within the program “Just Transition Fund” is evaluating and validating different carbonation techniques on mineral materials, such as concrete waste for the concrete industry. The high potential for permanent carbon sink and enhanced upcycling by accelerated carbonation on recycled concrete aggregate (RCA), provides a promising enhancer of aggregate value as carbonated recycled aggregate (CRA). In certain recycling processes and industrial actors, the treatment process undergoes an accumulation of RCA in intermediate storage facilities under seasonal climate conditions (sheltered) before utilization in new application. The objectives of this study were to investigate the effective CO<sub>2</sub> binding capacity on RCA (8/16 mm) preconditioned under those seasonal median temperatures (8 °C and 20 °C) and relative humidities (50 ± 5 % and 85 ± 5 %), denoted as 8C-RCA and 20C-RCA respectively, before being exposed to accelerated carbonation (10 % CO<sub>2</sub>-vol, 38° C, 97 % RH) under 4 hours, with typical flue gas parameters from the local municipal incineration plant. To quantify the bound CO<sub>2</sub> on such heterogenous material, a sample preparation method was investigated to retrieve a representable subsample using mechanical rotator dividers, followed by crushing to 0/4 mm and isopropanol exchange for hydrate stoppage. The crushed RCA into 0/4 mm was repeatedly subdivided in rotator for greater homogenization to about 50 grams and later milled for analyses. The analysis consisted of the thermogravimetric derivate curve for identification of the dehydrated and decomposition ranges of carbonates, starting from 500 to 800 °C. The decomposition interval was used for assessing the gravimetric measurement points on larger sample sizes (~15g x3) in a temperature-controlled oven. The results showcase the sample preparation having excellent coefficient of variance (C.o.V.) of less than 3% overall. With the overall CO<sub>2</sub> bound on 8C-RCA and 20C-RCA, the values before carbonation are about 1.45 % and 1.25 % respectively, indicating a higher natural carbonation before the accelerated testing on 8C-RCA. After four hours of accelerated carbonation, the CO<sub>2</sub> sequestered under conditions applied in the 20C-RCA case rose from 1.25 % to 2.51 % while in the 8C-RCA case, it rose from 1.45 % to 2.0 %. This difference could potentially be due to kinetics and transport properties from the different preconditioned RCAs prior to accelerated carbonation under those specific parameters.



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# Internal carbonation and hydration of cementless steel slag binder with the addition of sodium-based carbonates

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*Keywords: Steel slag; Internal carbonation; Hydration; Sodium-based carbonate*

Basic Oxygen Furnace (BOF) slag is a solid waste generated during the steel-making process, with approximately 200 million tons produced globally every year. It comprises hydraulic mineral phases such as belite ( $\alpha$ -C<sub>2</sub>S,  $\beta$ -C<sub>2</sub>S), brownmillerite (Ca<sub>2</sub>(Al,Fe)O<sub>5</sub>), along with almost non-reactive RO phase (MgO-FeO-MnO solid solution) and kirschsteinite, resulting in very low mechanical performance and hydration reactivity. To utilize these materials as high-value products, accelerated carbonation has been identified as a potential method for long-term CO<sub>2</sub> storage and enhancing product performance. As an internal CO<sub>2</sub> mixing method to accelerate the carbonation of steel slag, this study explores the effect of the addition of sodium-based carbonates on the carbonation and hydration of cementless slag systems. Specifically, sodium bicarbonate (NaHCO<sub>3</sub>), commonly known as baking soda, and sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>) are used. These carbonates readily dissolve in water, creating an alkaline environment and releasing carbonate ions. NaHCO<sub>3</sub> and Na<sub>2</sub>CO<sub>3</sub> are not only typical products or byproducts of carbon capture utilization and storage (CCUS) technologies but also effective CO<sub>2</sub> carriers for onsite carbonation, containing 52.4 g and 41.9 g of CO<sub>2</sub> per 100 g of dry powder, respectively.

BOF slag powder (< 75  $\mu$ m) was mixed with a water-to-solid ratio of 0.24 for microstructural analysis at 3, 7, 14, and 28 days (d). When 1% by weight of sodium-based carbonate was added to the slag, the carbonates identified were calcite, monocarbonate, and hemihydrate, along with the formation of katoite, portlandite (CH), and traces of vaterite and aragonite. Monocarbonate and hemihydrate were clearly observed at 3 d in the low-angle region of X-ray diffraction patterns, while Fe-containing AFm phases were formed between 14 and 28 d. It is expected that monocarbonate and hemihydrate could prevent system shrinkage and densify the microstructure due to their ability to chemically bind a significant amount of water molecules and their voluminous nature. Thermogravimetric analysis showed that chemically bound water (CBW) in plain slag paste increased from 3.11% to 3.74%, while it rose from 4.42% to 5.59% and from 4.36% to 5.24% with Na<sub>2</sub>CO<sub>3</sub>/NaHCO<sub>3</sub> addition, respectively. CH was completely consumed after 3 d, and hydration and carbonation continued with sodium-based carbonates added. While plain paste showed negligible change in chemically bound CO<sub>2</sub> (CBC) from 1.95% to 1.94%, CBC increased from 2.37% to 3.29% and from 2.38% to 4.07% with Na<sub>2</sub>CO<sub>3</sub>/NaHCO<sub>3</sub> addition, respectively. The result suggested that sodium-based carbonates reacted with the slag to form stable carbonate minerals. The 28-d compressive strength of the plain paste did not reach 3 MPa, but with the addition of sodium-based carbonate, it approached 20 MPa. Future research could focus on increasing sodium carbonate concentration to further improve CO<sub>2</sub> uptake and mechanical performance by tracking the long-term stability of carbonation products and phase composition.

## **Industrial experiences of enforced carbonation of recycled concrete paste**

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CO<sub>2</sub> mineralization technology using recycled concrete paste (RCP) as a sequestration substrate not only has the advantage of producing a new SCM with lower CO<sub>2</sub> footprint, but also complies with the circular economy concept.

The present work covers the upscaling of the enforced carbonation (EC) technology from laboratory to full scale, reporting the key learnings after the execution of industrial trial with different technologies at cement plant level.

After the laboratory development, the ideal conditions for enforced carbonation were well understood and with that, different SO<sub>2</sub> scrubbing technologies available in cement plants were identified to validate the laboratory findings in terms of kinetics and carbonated RCP (cRCP) performance. In the first campaign, tests were executed in a semi-dry scrubber at Brevik cement plant in Norway where the regular absorbent was exchanged for RCP, proving industrially that carbonation of RCP is feasible. The given scrubber could operate continuously in the optimal conditions. In addition to CO<sub>2</sub> mineralization, RCP was also found useful to sequester SO<sub>2</sub> and other pollutants. The analysis done in the produced cRCP allowed to estimate that 100 kg of CO<sub>2</sub> per ton of starting RCP were captured. This was achieved in less than 2 hours of stable operation and without a process optimization, proving that EC technology is industrially feasible. Additionally, the kiln stack analyzer showed that SO<sub>2</sub> emissions were reduced from 280 – 350 mg/Nm<sup>3</sup> to 60 – 80 mg/Nm<sup>3</sup>

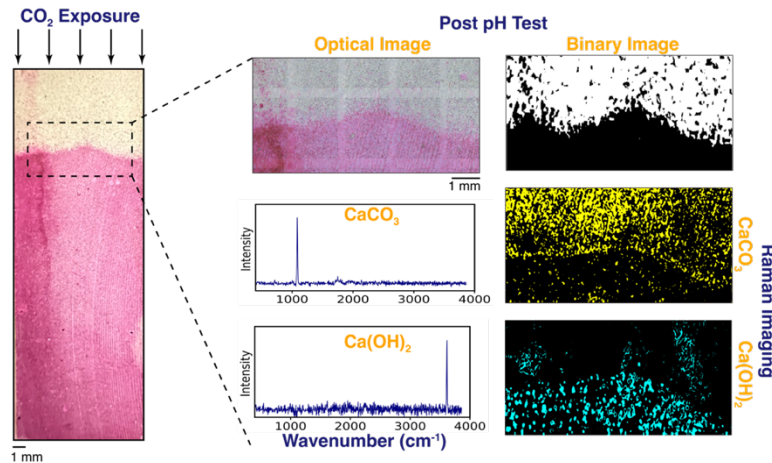
In the second campaign, a wet scrubber available at Ribblesdale cement plant was used for the same purpose, preparing a slurry containing RCP that replaced the standard absorbent. In this case the trial confirmed the fast kinetics of the wet carbonation process as the reaction was completed in a matter of minutes. The same final CO<sub>2</sub> sequestration rate as in Brevik trial was achieved. Finally, a ball mill designed for coal grinding that could be operated with kiln flue gases was successfully used to test a semi-dry carbonation of RCP to validate the concept of a reactive grinding, where continuously new surfaces are generated in the substrate to increase the process efficiency. In this case, RCP was fed to the mill instead of coal, and the process conditions were modified to maximize the retention time and relative humidity to allow carbonation to take place. In such setup, 70% of the carbonation potential was achieved, proving that this concept has a high potential to be upscaled and optimized due to its simplicity. In comparison to scrubbing technologies, a reactive grinding in a ball mill will provide the benefit of a simpler operation at lower specific energy consumption. Summarizing, the upscaling of EC of RCP has proven its promising features to be used as CCUS and allowed to identify the key aspects needed to develop and design the first-of-its-kind continuous demonstrator to be installed in a cement plant already this year.

# Tracking and Quantifying the Extent of Carbonation via Raman Imaging

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**Keywords:** Carbonation depth; confocal Raman imaging; image analysis; durability



Carbonation of cementitious systems is a growing field of study due to cement's potential to permanently store CO<sub>2</sub> via mineralization. Moreover, carbonation is a well-known degradation mechanism that lowers the pore solution pH, potentially promoting corrosion in reinforced concrete systems. Currently, several methods are available for recording the extent of carbonation and studying carbonation kinetics. These range from observing the surface pH change via a phenolphthalein indicator to quantifying calcite via bulk methods (XRD, TGA). However, there are fine microstructural details at the carbonation front that these traditional techniques may not be able to record. Recently, Raman imaging has been reported to be a potential tool for studying microscale precipitation and growth of calcium carbonate on cementitious substrates subject to carbonation.<sup>1</sup> We are now interested in exploring if this technique can be extended to track the extent of carbonation and if any new insights can be gained on the nature of the carbonation front.

In this study, we report our preliminary results with Raman imaging on a series of cementitious pastes subjected to CO<sub>2</sub> exposure. We subject prismatic specimens of varying porosity (w/c ratio ranging from 0.4 to 0.7) to unidirectional CO<sub>2</sub> ingress at 20% CO<sub>2</sub> concentration at a relative humidity of 60% and a temperature of 50 °C for 3 days. As expected, the sample's porosity directly affected the extent of carbonation, which was verified by surface pH change measurements conducted by the phenolphthalein indicator via optical imaging. However, the exact extent of carbonation, as measured by calcite precipitation via Raman imaging, portrays a slightly more complex picture than the sharp front often visible by the pH change measurements. In the talk, we will elucidate the nature of this complex microstructure modified and shaped by the active growth of calcite. In summary, optical imaging measurements, when combined with Raman imaging, offer a rich and detailed view of the complex, evolving carbonation front.

<sup>1</sup> S. Srivastava, N. Garg, Tracking spatiotemporal evolution of cementitious carbonation via Raman imaging, *J. Raman Spectrosc.* 54 (2023) 414–425. <https://doi.org/10.1002/jrs.6483>.

# Natural and enforced carbonation of hydrated cement paste

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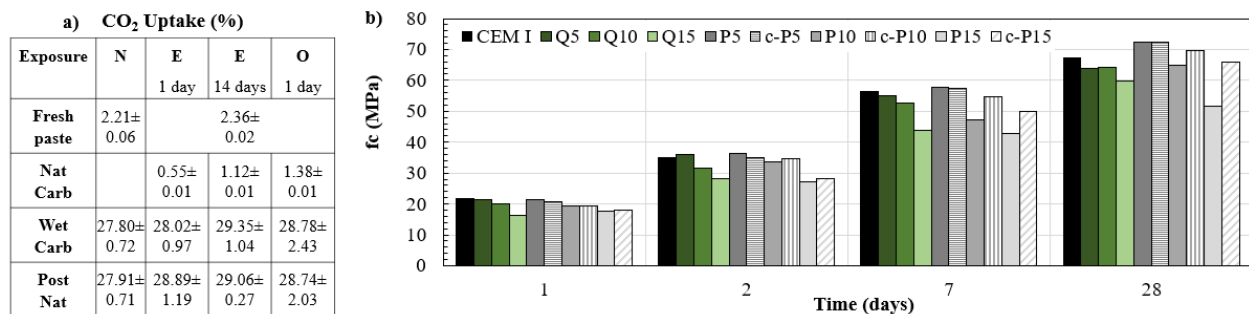
*Keywords: Natural and enforced carbonation; hydrated cement paste; mineral carbonation*

Mineral carbonation is a technology that enables the utilization of CO<sub>2</sub> into Recycled Concrete Fines (RCF). While this is an enforced process induced in a reactor, carbonation also occurs naturally. Our study helps distinguish between natural and enforced carbonation on hydrated cement Paste (P) by focusing on the quantification of a) P's natural and enforced carbonation and 2) the effect of enforced carbonation on the reactivity of carbonated paste (cP) as a cement replacement by compressive strength tests ( $f_c$ ).

Natural carbonation is studied by exposing hydrated cement paste (P) to: N – N<sub>2</sub> in a glove bag; E – lab controlled environment (CO<sub>2</sub>: 366±12 ppm, %RH: 26±9%, T: 23±0.3°C) for 1 and 14 days; and O – oven controlled environment (CO<sub>2</sub>: 153±17 ppm, %RH: 40±1%, T: 40±1°C) for 1 day. Next, all samples are carbonated (Wet Carb) in a reactor using a water/solid mass ratio of 10 and a gas flow rate of 2.0 L/min at 25% CO<sub>2</sub>. Then, the carbonated samples are exposed to E conditions (for 1 week) to check if additional carbonation occurs (Post Nat). Fig.1a shows the CO<sub>2</sub> uptake – normalized to the fresh paste mass (LOI: 27.5%). While limited to the evaluated cement paste and test conditions, our results highlight that:

- it is important to measure the CO<sub>2</sub> content of the fresh material prior to experiments to establish a baseline;
- P's CO<sub>2</sub> uptake due to natural carbonation is rather low (Max 1.38%±0.01), still must be prevented.
- CO<sub>2</sub> uptake from wet carbonation is not affected by either the starting state or natural carbonation (2 – 4%), since all tested exposure conditions yield similar final contents, i.e. 28.6%±0.6%; and
- no further natural carbonation – or a rather negligible amount – takes place after enforced carbonation;

Fig.1b shows the  $f_c$  results for binder systems P, cP, and quartz (Q), where CEM I 52.5N is replaced at ratios from 5 to 15% by weight of cement. The results indicate that, up to 28 days, replacing up to 10% of cement with cP does not compromise the cement's  $f_c$  performance significantly.



**Fig. 1.** Test results: a) CO<sub>2</sub> uptake and b) compressive strength ( $f_c$ )

To conclude, this study provides quantitative data on the natural carbonation that might occur during storage and experimental procedures. Our results suggests that proper assessment of the initial carbonate state of the fresh material is relevant for a reliable quantification of the enforced carbonation and control of measurements, keeping the CO<sub>2</sub> uptake baseline constant. Also, we identified that replacing up to 10% of CEM I with carbonated paste is practically achievable, opening venues for the use of carbonated RCF.

# Carbon sink role of lime-based construction units: Carbonation and time-dependent LCA study

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*Keywords: Carbonation, CO2 curing, lime, bricks, LCA*

This study investigates the CO<sub>2</sub> uptake kinetics and capacity of Autoclaved Aerated Concrete (AAC) blocks and Calcium Silicate Units (CSUs) through accelerated lab-based weathering conditions. The study encompasses chemical, mechanical and physical analysis of these materials after subjecting them to a climate chamber maintaining 20 % CO<sub>2</sub> air concentration, 50 % relative humidity and a temperature of 50 °C for varying time periods. Results show marginal changes in mechanical strengths, while significant improvement in water absorption properties after longer exposure times. To quantify the CO<sub>2</sub> absorption capacity and its impact on bricks properties, the degree of carbonation is calculated using thermogravimetry (TGA) measurements on samples collected destructively from specimen's cross sections at various depths of degradation.

Furthermore, the investigation delves into the broader context by exploring the potential carbon sequestration capability of these materials at a macro scale. This is accomplished by conducting a time-dependent Life Cycle Assessment study to gauge the carbon sink potential.

# CO<sub>2</sub>-induced Mineralogical Changes in Concrete Wash Water: Thermodynamic and Experimental Assessments

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*Keywords: Concrete wash water; CO<sub>2</sub> treatment; Thermodynamic modeling, X-ray diffraction*

The handling and disposal of concrete wash water (WW) is an operational, regulatory, and logistical challenge. In response, beneficiation of WW slurry through CO<sub>2</sub> treatment has emerged as a practical solution, allowing its reuse as mix water in concrete production. This study investigated the mineralogical changes occurring due to CO<sub>2</sub> treatment through a combination of thermodynamic and experimental approaches (XRD, TGA, pH, and ICP-OES). WW treated with increasing levels of CO<sub>2</sub> (5% to 25% by weight of cement at 5% intervals) were evaluated. The results indicate that the treatment is successful at accelerating the aging of the WW. In particular, the silicate clinker phases dissolve during the treatment to form calcite, amorphous calcium carbonate, and a low Ca/Si C-S-H gel. The aluminate phases do not participate during the treatment. This is related to a dissolution of the sulfate carrier during treatment without significant precipitation of sulfate-bearing phases, and hints toward a sulfate poisoning of the aluminate reactions. These results are confirmed by the solution analysis which shows that portlandite and ettringite becomes less stable during the treatment, while the low Ca/Si C-S-H becomes supersaturated. Therefore, the CO<sub>2</sub> treated WW is a more stable product, which could further contribute to the hydration of cement paste with both a filler effect from the calcium carbonate, and a pozzolanic effect from the low Ca/Si C-S-H gel. These findings contribute valuable insights to facilitate widespread adoption of CO<sub>2</sub> treatment as a beneficiation technology for WW slurry.

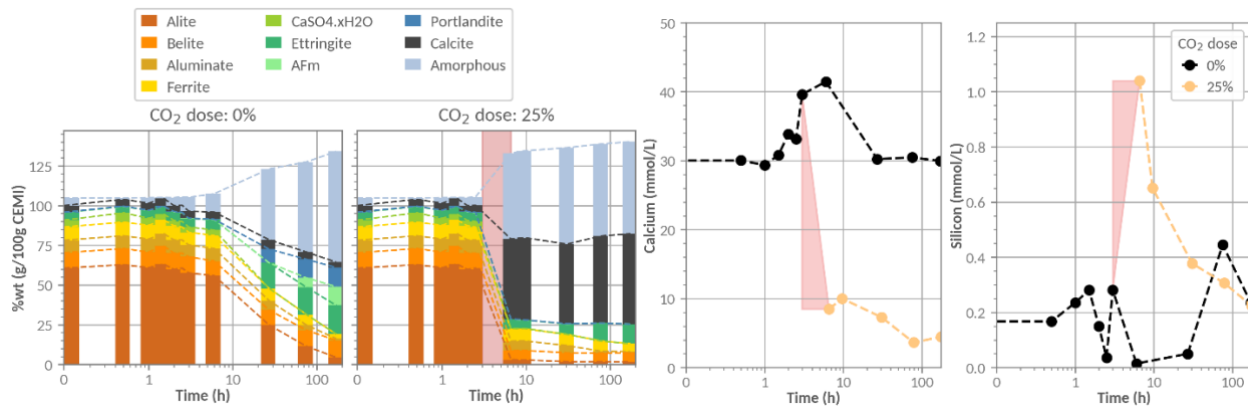


Fig. 1: Phase assemblages and, calcium and silicon concentration in the solution before and after the CO<sub>2</sub> treatment. The red areas represent the duration of the treatment.

# Characterizing the Pore Structure of CO<sub>2</sub>-injected Cementitious Systems

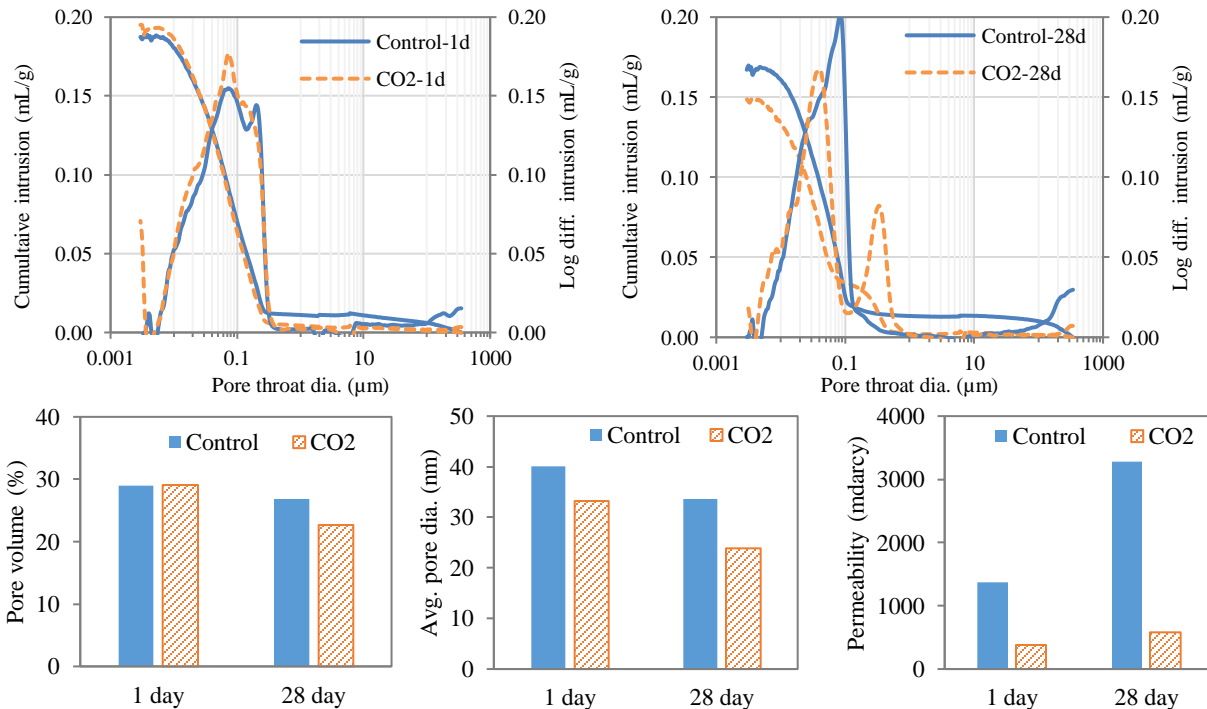
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*Keywords: Concrete; CO<sub>2</sub> injection; Pore structure, Mercury intrusion*

The pore structure of cementitious materials plays a crucial role in their mechanical properties and durability. While the beneficial effects of CO<sub>2</sub> injection (during concrete mixing) on the performance of cementitious systems have been well-documented, its influence on the pore structure remains largely unexplored. This study was aimed at characterizing the pore structure of CO<sub>2</sub> injected cement paste using the mercury intrusion porosimetry (MIP) technique. Cement paste samples were cast using ASTM C150 Type I/II cement and 0.3% CO<sub>2</sub> (by wt. of cement) at a water-to-cement ratio of 0.60. Samples were cured for 1-day and 28-day from which 5 to 15 mm small chunks were cored and then subjected to MIP. Fig. 1 presents the cumulative and log differential intrusion curves in top row while the calculated parameters are plotted in the bottom row. The intrusion curves of control and CO<sub>2</sub> samples were not significantly different at 1 day; however, 28-day curves revealed pore refinement occurring due to CO<sub>2</sub>-injection with reductions of around 15%, 30%, and 45% in the pore volume, average pore diameter, and threshold pore diameter, respectively. Furthermore, the permeability (calculated) was also significantly reduced, indicating improved resistance to fluid flow. These findings indicate that the precipitation of CaCO<sub>3</sub> due to CO<sub>2</sub>-injection refines the pore structure, potentially impacting (positively) the mechanical properties and durability of cementitious systems.



**Fig. 1.** Cumulative and log differential curves of Control and CO<sub>2</sub>-sample obtained from MIP (top row) and calculated pore structure parameters (bottom row)

# Advanced carbonation technologies for total recycling of concrete wastes

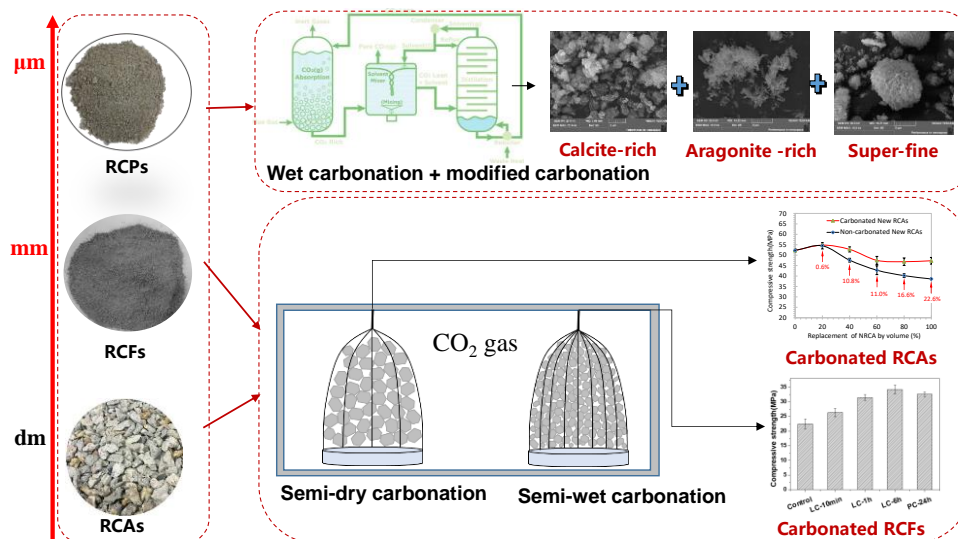
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*Keywords: Concrete waste; carbonation technologies; Recycling; Mechanisms*

The whole world is facing a critical environmental challenge due to the extensive release of CO<sub>2</sub> and the construction sector plays an important role. Carbonation of waste concrete could address the CO<sub>2</sub> release issue. Our recent research has shown that water plays an important role in facilitating gas dissolution, ion species transformation, and the formation of carbonation products in all carbonation reactions. Based on the role of water, three different advanced carbonation technologies to achieve the total recycling of concrete waste. Advanced technologies including pressurized semi-dry carbonation, flow-through semi-wet carbonation and wet carbonation methods were developed to treat concrete wastes with different sizes. Recent results indicated that both carbonation efficiency and carbonation products were significantly influenced by water content, and waste concrete can be turned into low-carbon products, including carbonated coarse aggregate (CRCA), carbonated fine aggregates (CRFA) and functional carbonated recycled concrete powder (CRCP). In detail, the performance of CRCA and CRFA can be significantly improved with after semi-dry carbonation and semi-wet carbonation for a short period, which can be used to replace natural aggregates. In addition, different kinds of CRCP including calcite-rich, micro-fibre-rich and superfine powders were produced. This CRCP not only was a highly active pozzolanic material but also an effective accelerator affecting the early hydration kinetics of OPC paste, thereby leading to an increased early compressive strength and a comparable 28-d compressive strength, even when 20% OPC was replaced by the CRCP replacement ratio. Furthermore, the high-performance concrete incorporating these carbonated concrete wastes can be produced, which not only promote the recycling of wastes but also sequestrate a large amount CO<sub>2</sub>. Overall, the results presented in this study suggested the provided carbonation technologies of concrete waste can make great contribution to the waste recycling and reduction of CO<sub>2</sub> emission of construction sector.





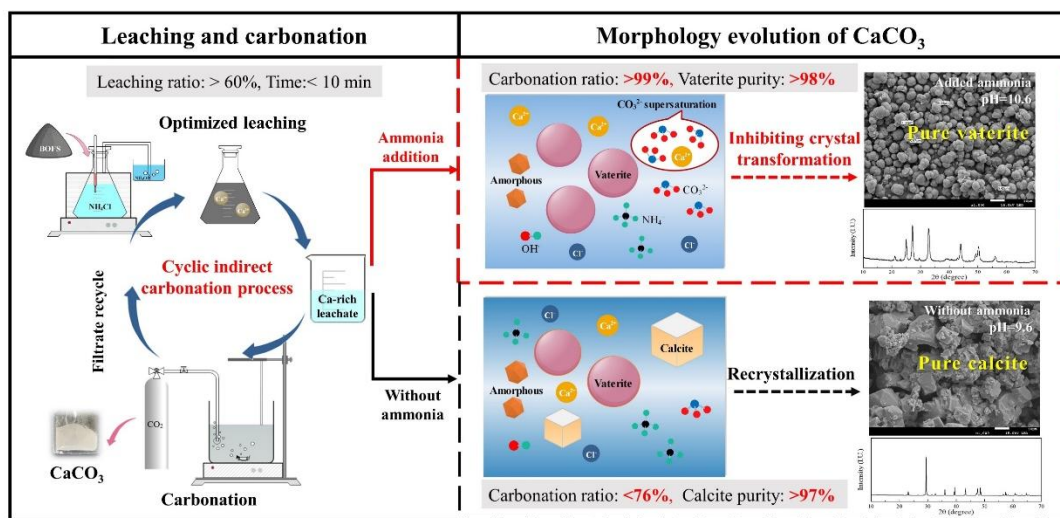
# Synthesis of pure vaterite via leaching-carbonation of basic oxygen furnace slag

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*Keywords: Indirect carbonation; leaching-carbonation process; basic oxygen furnace slag; vaterite; stabilization mechanism*



Carbonation of steel slag such as basic oxygen furnace slag (BOFS) is one of the most promising ways for effective CO<sub>2</sub> sequestration and enhanced BOFS utilization. In-situ formation of carbonation products can mobilize the application of BOFS for use in construction materials. Against the backdrop of the limited industrial application and low added value of PCC produced through the indirect carbonation of BOFS, this study proposed a simple approach (extracting Ca<sup>2+</sup> from BOFS using ammonium chloride (NH<sub>4</sub>Cl) and subsequently optimizing the aqueous carbonation process) for synthesizing high-purity vaterite with greater added value. Meanwhile, the crystal transition and stabilization mechanism were analyzed. The optimal conditions for calcium ion leaching were first identified to yield a 60.3% Ca<sup>2+</sup> extraction in 10 min, and the proposed high-order mass-loss method model showed good agreement with Ca<sup>2+</sup> leaching. pH ~ 10.6 of the solution was refined using ammonia to achieve a higher CO<sub>3</sub><sup>2-</sup>/Ca<sup>2+</sup> ratio for the stimulation of vaterite nucleation. Also, the presence of NH<sub>4</sub><sup>+</sup> and metal ions from BOFS acted as crystalline control agents, impeding the transformation of vaterite into calcite. Overall, the parameter designed in this study can achieve 99.5% carbonation conversion with a CO<sub>2</sub> uptake of 185 kg for one ton of BOFS used, yielding a high purity (>98%) homogeneous vaterite. In addition, the carbonation filtrate can be recycled up to six times without compromising the efficacy of the process or the crystalline integrity of the carbonated product. The solid residue, composed mainly of silica gel and inert RO phase, exhibits good pozzolanic reactivity for use as supplementary cementitious materials. Findings from this study provide new insights into the valorizing of BOFS to mitigate CO<sub>2</sub> emissions and promote the sustainable industrial application of the BOFS indirect carbonation process.

# Composition and thermodynamic properties of the alumina-silica gel formed upon aqueous carbonation of C-S-H and ettringite

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## ABSTRACT

The amorphous alumina-silica gel, produced through enforced carbonation of recycled concrete fines from end-of-life concrete, exhibits rapid pozzolanic properties as an SCM in composite cements. It contains different types of SiO<sub>4</sub> species, with AlO<sub>4</sub> sites distributed in the aluminosilicate network. The objective of this study is to determine the maximum aluminum uptake in the alumina-silica gel, and thereby its boundary composition, and to establish a thermodynamic model for the alumina-silica gel. The latter will be useful for predictions of the impact of parameter variations and compositional changes under different carbonation conditions and for different starting materials.

Aqueous carbonation of synthesized C-S-H phases and ettringite is used as a model system for the carbonation of hydrated Portland cement. Synthesized C-S-H with Ca/Si ratios of 0.83 and 1.4 were prepared and mixed with synthesized ettringite. The mixtures were carbonated for 6 hours under wet conditions and studied by *in-situ* pH, TGA and <sup>27</sup>Al, <sup>29</sup>Si NMR measurements. After carbonation, the aluminum from ettringite is preferentially present in tetrahedral coordination in the alumina-silica gel. However, a secondary alumina-gel containing aluminum in octahedral coordination Al is also present and the fraction of this gel increases with increasing ettringite content of the C-S-H – ettringite blends (e.g., from 1 wt% to 20 wt% as investigated here). The fraction of alumina gel is less affected by the Ca/Si ratio of the C-S-H phase used in the experiments.

A thermodynamic model for the alumina-silica gel is established based on different compositions of alumina-silica gels formed in cementitious systems, using the GEMS 3.9.6 software and the Cemdata 18.1 database. The solubility products, determined by GEMS, show a decreasing linear relationship with the Al/(Al+Si) ratio of the alumina-silica gel over the range from 0 to 0.5. This suggests that the gel can be stabilized by an increasing amount of AlO<sub>4</sub> sites.

**KEYWORDS:** Carbonation, alumina-silica gel, thermodynamic modeling, NMR spectroscopy.

# Effects of Relative Humidity and Temperature on Carbonation Dynamics of CaO and Ca(OH)<sub>2</sub>

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*Keywords: carbonation, construction materials, kinetics*

The carbonation of mineral oxides/hydroxides like lime and slaked lime holds promise for the creation of net-neutral or net-negative carbon construction materials, providing a sink for atmospheric CO<sub>2</sub> in the process. The carbonation extent and kinetics are influenced by factors equally dependent on the base material physicochemical properties and the conditions employed for carbonation. Optimizing these conditions and understanding the instantaneous carbonation mechanisms are the premises for a fast carbonation process.

In this work, we tested the hydration of CaO and subsequent carbonation at various relative humidities and temperatures. Higher relative humidity (~60%) and higher temperature (40 °C) can improve the carbonation degree. However, high levels of relative humidity and temperature influence the diffusion and solubility of CO<sub>2</sub>, respectively. The carbonation mechanisms were studied by in situ Raman during the reaction process. This study provides detailed methodologies for studying the carbonation process and unveils the reaction mechanism, which is of significant importance for both academic and practical applications.

# Principle of a new method to determine the CO<sub>2</sub> binding capacity of recycled concrete aggregates

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*Keywords: carbonation, recycled concrete aggregates, CO<sub>2</sub> uptake, test methods*

In the scope of Carbon Capture, Utilization and Storage (CCUS), carbonation of recycled concrete aggregates (RCA) is one way of sequestering CO<sub>2</sub> from industrial facilities as well as atmospheric CO<sub>2</sub>. This kind of process can be seen as a low-cost and low-tech solution. The effectiveness of such a solution depends mainly on the CO<sub>2</sub> binding capacity of RCA. Thus, test methods are needed to assess it properly. For this purpose, thermogravimetric analysis (TGA) is a usual method used to quantify the amount of CO<sub>2</sub> bound by carbonation. However, TGA has limitations due to the small sample size (a few hundred mg), which requires careful sampling and preparation. In the present paper, we introduce the principle of a new method allowing for testing larger masses of RCA. This method consists in determining instantaneous CO<sub>2</sub> binding rate (IBR). A sample of RCA is placed in a closed cell where the CO<sub>2</sub> concentration is monitored by a sensor. As the CO<sub>2</sub> is consumed during carbonation of the sample, regularly, the cell is refilled by CO<sub>2</sub> to keep the initial concentration. IBR are deduced from the decreases of CO<sub>2</sub> concentration. The quantity of bound CO<sub>2</sub> is calculated by integrating the time-evolution of IBR. Based on the same principle, two versions of this test method are available. The first one is manual and deals with atmospheric carbonation (Figure 1). The second one is automated and simulates an accelerated carbonation at regulated high CO<sub>2</sub> concentration. To validate the proposed methods, samples of crushed mortar were tested, and the CO<sub>2</sub> binding capacities obtained through different methods, IBR, TGA and loss on ignition (LOI), were compared.

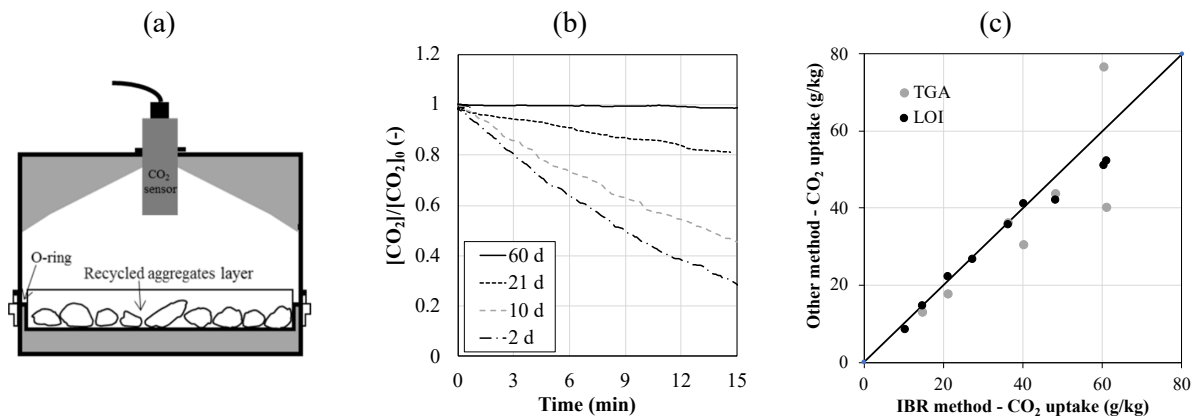


Fig. 1. Atmospheric carbonation of RCA: (a) scheme of the IBR method device, (b) evolution of the CO<sub>2</sub> concentration related to the initial concentration in the IBR cell for a crushed mortar sand after different carbonation durations, (c) correlation between results from 3 methods (uptake in g of CO<sub>2</sub> par kg of RCA).

# Investigation into using CCU material by-produced from waste concrete sludge in concrete

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*Keywords: Sludge; calcium carbonate; concrete; CCU materials*

Japan has joined the global initiative and set an objective of reducing CO<sub>2</sub> and achieving a carbon-neutral society by the year 2050. This paper presents the technology for the production of Carbon Capture and Utilization (CCU) materials for use in the construction industry derived from waste concrete sludge. As illustrated in Figure 1, the waste concrete sludge is first diluted to promote leaching of calcium ions into the liquid following which it is filtered using a filter press. Boiler exhaust gas is bubbled into this filtrate, producing fine particles of calcium carbonate called "Ecotancal" and preventing boiler gas from escaping into the atmosphere. The remaining solid by-product also contains calcium ions in the form of cement hydration products, which on crushing and carbonation yields a fine powder called "DACS<sup>®</sup>". In this report, the effect of adding both "Ecotancal" and "DACS<sup>®</sup>" as mineral admixtures in concrete is discussed.

Non-Dispersive Infrared analysis of Ecotancal shows that it consists of over 90% by mass of calcium carbonate (CaCO<sub>3</sub>), and it has a BET-specific surface area six times that of Portland cement. Overall, the CO<sub>2</sub> absorbed by the filtrate outweighs the CO<sub>2</sub> emitted during the production of Ecotancal, making it carbon negative by -390 kg-CO<sub>2</sub>/t. Since Ecotancal is primarily calcium carbonate, it can be used to enhance the cohesiveness of Self-Compacting Concrete (SCC). The effect of replacing a part of cement with Ecotancal on the plastic viscosity of mortar was investigated using a Rheometer. The results shown in Figure 2 indicate that as the replacement ratio increases, the plastic viscosity increases indicating the addition of Ecotancal enhances the cohesiveness of mortar.

The production volume of DACS<sup>®</sup> is about 40 times that of Ecotancal and TG-DTA substantiates that it can contain up to 35% by mass of calcium carbonate after carbonation. To investigate the effect of adding DACS<sup>®</sup>, compressive strength tests were conducted on mortar specimens with 50 kg/m<sup>3</sup> and 100 kg/m<sup>3</sup> of DACS<sup>®</sup> as a replacement for Sand. The results shown in Figure 3 indicate that replacing sand with DACS<sup>®</sup> gives higher 7-day and 28-day compressive strength.

In conclusion, this report presents the technology for the production of two types of CCU materials from waste concrete sludge and highlights the benefits of adding them to mortar.

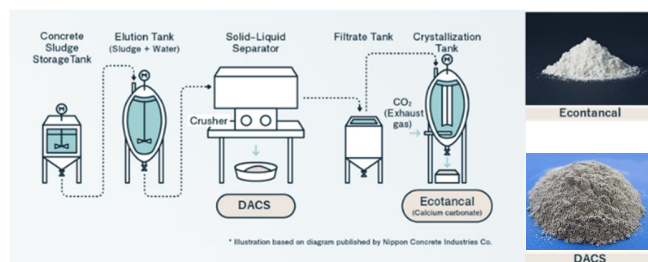


Fig. 1: Process flow for production of Ecotancal and DACS

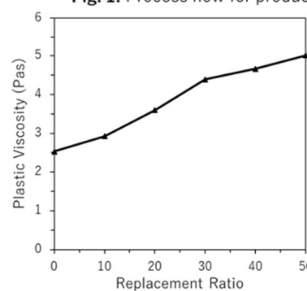


Fig. 2: Improvement in Cohesiveness on adding Ecotancal

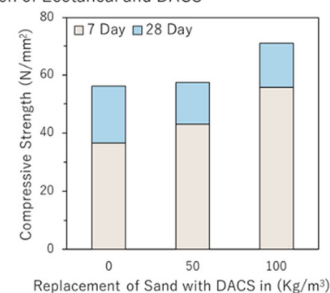


Fig. 3: Increase in compressive strength on replacing sand with DACS

# Hydration and Carbonation of Lime-cement binders in Carbonation Curing Environments

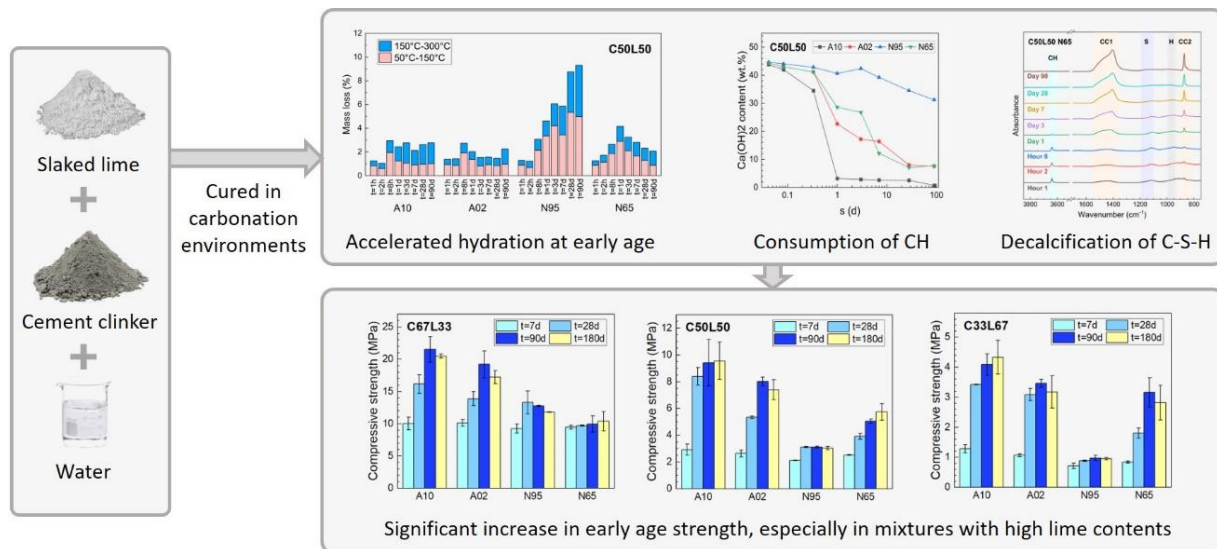
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*Keywords: lime-cement binders, hydration, carbonation*

Lime-cement binders get hardened through both hydration and carbonation reactions, but interactions between these two mechanisms can be complex in carbonation curing conditions. While hydration products like  $\text{Ca(OH)}_2$  and C-S-H could get carbonated,  $\text{CaCO}_3$ , the primary carbonation product, could also affect hydration kinetics and hydration product composition. Therefore, it is necessary to understand the whole hardening process and clarify influences of key factors like lime-to-cement ratio ( $l/c$ ) and  $\text{CO}_2$  concentration. Towards this goal, five different mixtures (i.e.,  $l/c=0.0, 0.5, 1.0, 2.0$  and Infinite) were exposed to three different carbonation curing environments (i.e., room conditions, accelerated carbonation environments with  $\text{CO}_2$  concentrations of 0.2% and 1.0%). Powder samples were collected from thin disk specimens for chemical analyses, and prism specimens were prepared for strength and carbonation depth tests. Experimental results show that the additional lime in lime-cement binders reacts with  $\text{CO}_2$  first. Nevertheless, carbonation of C-S-H was still observed when there was uncarbonated  $\text{Ca(OH)}_2$  in the system. This phenomenon is more significant in samples exposed to accelerated carbonation environments. In those groups,  $\text{Ca(OH)}_2$  and  $\text{CaCO}_3$  contents change marginally after 3 days, suggesting that rapidly formed carbonation products could hinder further carbonation of lime, and thus weaken the protective effect of lime. Due to the high porosity of lime-cement binders, carbonation products can only cover the surface of lime particles, but cannot block all the pores. Therefore, the transport of  $\text{CO}_2$  is still accelerated in  $\text{CO}_2$ -rich environments, and greater carbonation depths were detected. However, influences on the compressive strength are complex. Though newly formed  $\text{CaCO}_3$  makes the microstructure denser, decalcification of C-S-H has negative impacts on mechanical properties. As a result, significant increases in the compressive strength were only observed in mixtures with high lime contents.



# Comparative studies on the methods to evaluate carbonation degree and activity of calcium-rich solids

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*Keywords: calcium-rich solids; carbonation degree; carbonation activity; evaluation methods*

The carbonation degree determines the carbonation capacity of materials and the actual amount of CO<sub>2</sub> absorption. Accurate and efficient assessment of the carbonation degree of materials can provide guiding suggestions for the selection of materials in actual carbonation applications and the optimization of carbonation environmental conditions. Therefore, methods to evaluate the carbonation degree were analyzed and compared from the perspectives of carbonable reactant consumption, product generation and mass change during carbonation in this study. Using these methods, the carbonation degree of steel slag, C<sub>3</sub>S and calcium carbide slag at l/s = 0 and l/s = 0.15 was measured by QXRD, TG or weighing.

The results demonstrated a similar variation trend in the carbonation degree of the three raw materials under different liquid-solid ratios, with a variance of ±1.07%. In addition, both XRD and TG analyses yielded highly comparable carbonation results, exhibiting linear fitting correlation coefficients greater than 0.98. However, slight differences were observed in the carbonation values obtained by the dry mass change method, particularly in the presence of liquid water. Nevertheless, all methods investigated in this study exhibited strong consistency and reliability, including the dry mass change method which displayed an acceptable deviation from other methods and was quickly and conveniently testable. Furthermore, a carbonation degree of 24 hours at l/s = 0.15 is proposed as an index to evaluate carbonation activity due to its ability to rapidly and effectively difference between different materials' carbonation potential. Finally, carbonation activity inventory was established, in which calcium-rich materials were categorized into five groups in terms of their activity. These evaluation methods for carbonation are conducive to select highly active materials and are expected to support improvements in environmental benefits associated with material carbonation treatment.

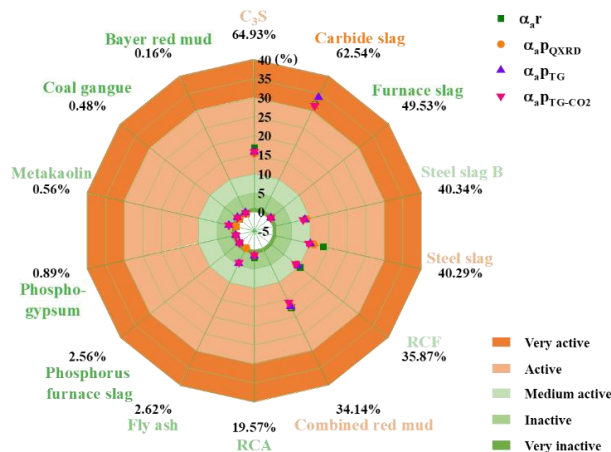


Fig.1. Carbonation activity inventory of calcium-rich materials.