

IMPERIAL

Carbon Sequestration in the Built Environment – Year 1 Report

CCUS Innovation 2.0

Key Knowledge Deliverable 6.1

November 2024

Key Knowledge Deliverable Cover Sheet

This Key Knowledge Deliverable (KKD) has been produced by Imperial College London as part of the Department for Energy Security and Net Zero £1bn Net Zero Innovation Portfolio (NZIP) - CCUS Innovation 2.0 programme. The document is reflective of the status of the project at the time of writing. The material presented could have been subject to change as the project matured. These documents should not be considered a full representation of the final project.

Project Description

This project seeks to further develop and scale a new carbon sequestration process which transforms waste CO₂ gas from industrial facilities into valuable construction products. Sequestered CO₂ through this process is cheaper than conventional approaches that rely on purification, liquification and offshore or geological storage. The CO₂ is stored in the form of a stable mineral which ensures they will be no leakage over time.

The patent-pending technology involves taking globally abundant magnesium silicate minerals and splitting this into magnesia and silica components. Through simple chemical processing two products of high purity are created: a) an amorphous silica that can be used as supplementary cementitious material (SCM) to facilitate low-carbon concrete and b) a concentrated magnesium solution in which CO₂ from industrial flues can be sequestered to produce other construction materials.

This CCUS Innovation 2.0 award will be used to increase our technology and commercial readiness level by de-risking and facilitating the development of a pilot facility, in order to demonstrate that the technology is economically viable and deployable at scale.

Description of the KKD

Progress during the first year of the project is reported, including key results from the deliverables completed to date. Changes to the original work programme are highlighted, and an explanation given as to why these were necessary. The project aims to develop a new carbon capture utilisation and storage (CCUS) process that is unique, because it captures CO₂ emissions directly from industrial sources, while also producing valuable low-carbon construction products. The costs of applying simple chemical processes to capture CO₂ emissions directly are far lower than direct air capture, liquefaction, or transport offshore, and this technology has the added benefit of producing construction materials. This means that the process developed has significant potential to be effective and commercially viable.

KKDs to be released in full

- D3.4 – Concrete Trials 3
- D4.4 – Product Optimisation 2

KKDs to be released after redactions

- D1.1 – Flue Gas Recovery and Testing 1
- D1.2 – Dissolution Procurement
- D1.3 – Dissolution Operation
- D1.4 – Flue Gas Recovery and Testing 2 & Carbonation Procurement
- D1.5 – Carbonation Operation
- D2.3 – Reagent Regeneration Procurement
- D2.4 – Reagent Regeneration Operation
- D3.2 – Concrete Trials 1
- D3.3 – Concrete Trials 2
- D4.2 – Process Optimisation
- D4.3 – Product Optimisation 1
- D5.2 – Business Development 2 (Supply Chain)
- D5.3 – Business Development 3 (Business Planning)
- D5.4 - Business Development 4 (Commercial Readiness)
- D6.1 – Year 1 Report
- D6.2 – Year 2 Report



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Any enquiries regarding this publication should be sent to us at:
nzip@energysecurity.gov.uk

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Results from the work to date

Deliverable 2.1: Reagent regeneration trials

[Deliverable Report not to be released]

Deliverable 3.1: Silica post processing trials

[Deliverable Report not to be released]

Deliverable 1.1: Flue gas recovery and testing

Carbonation in this process is a liquid-gas reaction between activated Mg and the CO₂ present in a flue gas. The activated Mg solution can contain residual Fe. This does not influence the rate of CO₂ sequestration and does not change the type of magnesium carbonate (MgCO₃) formed, and the MgCO₃ product has properties independent of the iron content in the starting magnesium silicate mineral. Four gas compositions were trialled at lab-scale. These were compressed air, pure CO₂, and two simulated flue gas streams. The rate at which CO₂ mineralises is determined by the rate CO₂ dissolves into the Mg solution. Carbonation has been investigated at three different CO₂ concentrations, 0.4, 15 and 100 %. Trials using 0.4 % (400 ppm, atmospheric concentration) did not produce any precipitation after 10 days of carbonation. Further research therefore focussed on using pure CO₂ and simulated flue gases. The rate of carbon mineralisation has been investigated at different magnesium concentrations. The concentrations studied were 0.1, 0.2, 0.3, 0.4 and 0.5 mol/L and pure CO₂ was used in these studies. Increasing the concentration of Mg in the solution increased the carbonation rate. At high concentrations the amount of precipitate formed thickens the slurry and reduces efficiency, reducing carbonation. Initial rate measurements for a 2.0 M Mg solution gave a value of 158 kg/m³/hr, and this is ~ 50 times the rate of sequestration in direct air capture, for a given volume. The observation that the capture efficiency is unaffected by the concentration of the CO₂ suggests that utilising multiple passes would capture significant proportions of CO₂. It is also worth noting that these numbers represent sub-optimised equipment with no pressurisation.

The magnesium carbonate produced has been characterised. The chemical species present in the flue gas do not have a direct impact on the morphology of the carbonate produced. Carbonate particles from simulated flue gas are consistently larger, owing to a longer reaction time. Slower growth crystals will be larger, and a long residence time allows for mechanisms such as Ostwald ripening to coarsen particles. X-ray diffraction showed that despite high

concentration of SO_x and NO_x in the simulated flue gas, the only insoluble phase present in the carbonate product was magnesium carbonate. Magnesium sulphate salts are present due to partially incomplete reactions, and ammonium sulphate can precipitate out of solution by drying. However, both are easily removed by washing. XRD analysis also showed that despite altering the gas composition, the magnesium carbonate formed was nesquehonite.

Deliverable 4.1: Materials Optimisation

[Deliverable Report not to be released]

Deliverable 5.1: Business development 1

[Deliverable Report not to be released]

Deliverable 3.2: Concrete Trials 1

The aim of this work was to assess the performance of the produced silica as an SCM, with the aim of producing low-carbon concrete. This process can produce silica SCMs with different purity levels, depending on the raw materials used. The work reported here aimed to assess the inherent characteristics of high- and low-grade silica samples. These were optimised for use as an SCM, and the performance of resulting concretes was benchmarked against Portland cement concrete and blended cement concrete using fly ash.

The work demonstrated that the silica SCM has higher pozzolanic activity than fly ash. This high reactivity causes rapid sulfate depletion, and this inhibits dissolution of the silicate phase, tricalcium silicate (C3S), adversely influencing the setting behaviour of paste samples. The high-grade silica is more reactive than low-grade silica, and the lower compressive strengths observed was due to problems with mixing the silica SCM, rather than reduced reactivity.

Deliverable 1.2: Dissolution equipment procurement

The dissolution stage in this process uses acids to digest olivine, releasing Mg and silica species. The activated Mg solution can contain residual Fe. Co-precipitation of Fe and silica produces a silica SCM with improved workability. This work specified and obtained the equipment required to operate dissolution and separation at increased scale. Additionally,

preliminary work was conducted to ensure that the reaction products are of target purity and reaction efficiencies are acceptable at increased volumes.

The necessary equipment has been purchased. Operating dissolution at an increased volume appeared to take longer than at lower volumes. The increased size of the glass beaker and an insufficiently powered hotplate may have caused a decrease in temperature and therefore reaction rate. Additionally, dissolution using salt precursors is slower than using the catalyst material. The reaction was stopped after three and a half hours. The initial solution was a dark green/orange due to a mixture of iron(II) and iron(III) ions being present. On addition of olivine, the solution becomes turbid and dark orange due to rapid oxidation of iron(II). The slurry gradually becomes yellow as the spent catalyst crystals precipitate and the solution loses the majority of its chromatic iron species. After dissolution is complete, the spent catalyst crystals are removed by filtration. The supernatant contains the $(\text{Mg,Fe})\text{SO}_4$ and silica species. In a complete reaction the supernatant would be a blue-green colour.

Deliverable 1.4: Flue gas recovery and testing 2 and carbonation procurement

A cylindrical continuous carbonation reactor has been developed. This allows 3L of MgSO_4 solution to react with different CO_2 containing gases. To start the experiment ammonia and MgSO_4 pumps are switched on while simultaneously opening the exit and inlet gas flowrate valves. As the first residence time passes the exit flow was collected and disposed of because the exit stream had a variable output flow residence time. The first residence time batch was used to achieve a continuous equilibrium, with the exit flow having the same residence time. Samples were taken at different intervals to see the effect of residence time on conversion. Samples were taken from 5 holes on top of the reactor during the second residence time. The inlet and outlet flows were stopped after the second residence time. The samples collected were filtered using a syringe filter and diluted by a factor of 10. The solution collected in the beaker for the duration of the second residence time was then left for an hour in the beaker before being vacuum filtered, dried at 60°C and in a furnace. The filter cake is then weighed and characterized using XRD, TG and SEM. Titration tests were conducted to estimate how much magnesium ion is in the solution to estimate the conversion at different residence times.

Deliverable 2.2: Reagent regeneration engineering design

[Deliverable Report not to be released]

Deliverable 4.2: Process Optimisation

The carbon mineralisation process produces nesquehonite, ($\text{MgCO}_3 \cdot 3\text{H}_2\text{O}$), a metastable magnesium carbonate phase. Over time, nesquehonite decays to produce hydromagnesite, ($\text{Mg}_5(\text{CO}_3)_4(\text{OH})_2 \cdot 4\text{H}_2\text{O}$) through a dissolution and reprecipitation reaction. This transition can be accelerated through the addition of water and elevating the temperature above 60°C . It is reported that this transition is thermodynamically favourable above 50 to 60°C . The morphological changes that accompany this transition are responsible for the setting mechanism of the material. The aim of this deliverable was optimisation of the brick and board production process.

Both block and boards have been successfully produced. A water to binder ratio of 0.6 gave the best performance. Curing at 60°C produced the best materials, although more testing is required at intermediate temperatures to determine the best curing temperature to use. Time at elevated temperature for curing had minimal impact on 28-day compressive strength data and a strong relationship between exists between the degree of transformation from nesquehonite to hydromagnesite and compressive strength.

Deliverable 5.2: Business Development 2 (Supply Chain)

Interactions with predominant European suppliers of Olivine are reported. Approximate quotes on bulk price and volumes available have been fed through into subsequent techno-economic analyses (TEA), incorporating scenario and sensitivity analyses. Where appropriate, reasonable assumptions have been made to complete the TEA.

Key conclusions are that olivine suitable for this carbon mineralisation process can be sourced from multiple quarries across Europe for use locally, or shipping to the UK. Sufficient capacity exists for short-term pilots and scaling of the mineralisation process, but further capacity would need to be built up to maximise the long-term CO_2 mitigation potential of the technology. Olivine can be affordably supplied from either Åheim or Chalkidiki to a UK-based pilot/industrial facility. The effects of transport on the emissions assessment of the carbon mineralisation process will need to be considered separately. Within the current set of modelling assumptions, the mineralisation process is economically viable, if olivine can be sourced, delivered, and milled for less than $\text{£}80$ per tonne.

Changes to the original work programme and project

D1.2 (Dissolution procurement) was moved back until sufficient completion of D2.2 (Reagent regeneration engineering design). This also had a knock-on effect on D1.3 which is dependent on the completion of D1.2. Subsequent Deliverables in WP1 (D1.4 and D1.5) are not dependent on the completion of D1.3, so were unaffected by this change. No Deliverables in any of the other WPs were affected by this change.

The subcontractor for D2.2 (Reagent regeneration engineering design) will be changed from Centre for Process Innovation (CPI) to Xytel Corporation. There will be no change to project costs, timelines, or outputs. Xytel Corporation has greater technical capacity and expertise in process engineering, particularly with scale up and the design of larger pilot and industrial facilities. Switching to Xytel at this stage will simplify future development work, removing the need for duplication and accelerating the route to commercialisation.

Seratech has become a Project Partner. Seratech is the spin-out company from Imperial College London that will commercialise the technology from this CCUS2 project. The company has negotiated an exclusive license with Imperial College London to commercialise IP developed by the team, including that arising from this project. The addition of Seratech as a Project Partner will allow it to utilise publicity arising from the project, and to build a stronger link between the technology, industry, and the public. This will greatly improve the commercial traction that Seratech is able to achieve and maximise the impact of this CCUS2 funding. At the time of application in November 2022, Seratech had not yet spun out from Imperial, so could not then be involved as a Project Partner. It has now spun-out as of April 2024.

Conclusions

This DESNZ funding has allowed significant progress on the development of an important carbon sequestration technology. This would not have occurred without this funding. An appropriate team with complimentary expertise has been assembled during the first year. An extensive amount of work has been completed to date, and this has benefitted from the excellent UK funded facilities at Imperial College London. Olivine is a globally available mineral with significant potential to contribute to carbon sequestration. The silica extracted from this in has great potential to be used as a supplementary cementitious material that can produce low-carbon cement. This is now widely regarded as a world leading development in delivering carbon negative concrete and infrastructure. Carbon sequestration occurs through the production of nesquehonite. The research completed to date shows that this can be used to manufacture hydromagnesite blocks and boards. This is critically important because of the relative amounts of silica and magnesium produced. Scope for additional innovation and IP is significant, and there is every potential for Seratech to become a world leading UK low-carbon company.

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