IMPERIAL

Product Optimisation 1

CCUS Innovation 2.0

Key Knowledge Deliverable 4.3

October 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 KKD

Report detailing the complete experimental plan for the remainder of this work package and intermediate results. The testing will include strength and durability results of magnesium carbonate products manufactured via the route(s) determined in D4.2 with various additions of filler and fibre materials to optimise performance.

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

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

Contents

Introduction

Background

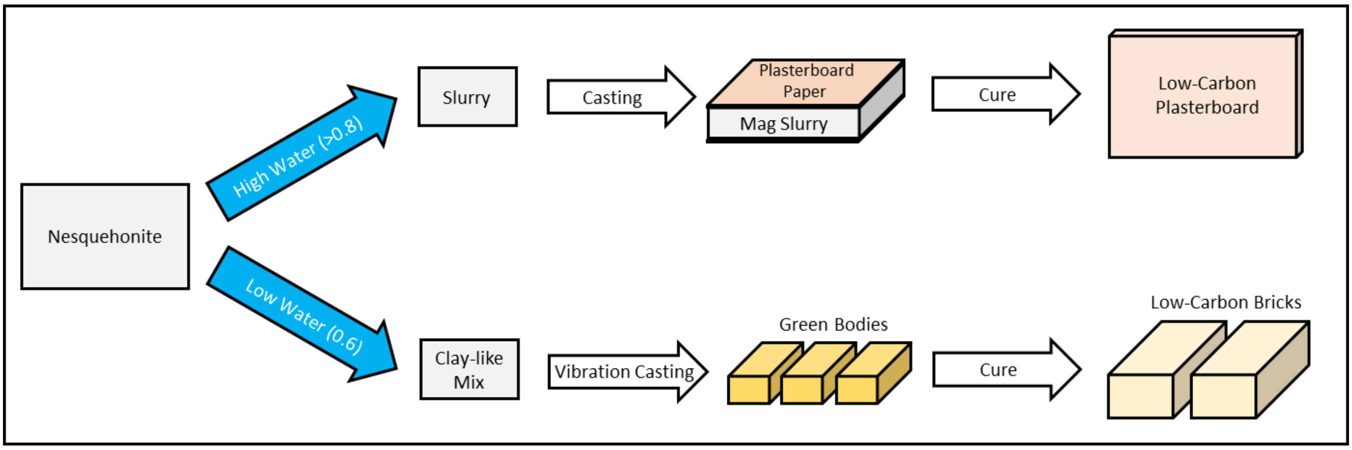
The carbon mineralisation process used in this project, produces nesquehonite, NQ, ($MgCO_3.3H_2O$), a metastable magnesium carbonate phase. With time, nesquehonite decays to produce hydromagnesite, HM, ($Mg_5(CO_3)_4(OH_2).4H_2O$) through a dissolution and reprecipitation reaction, seen in Eq. 1.

$$5MgCO_3.3H_2O \rightarrow Mg_5(CO_3)_4(OH_2).4H_2O + CO_2 + 10H_2O \qquad \text{Eq. 1}$$

This transition can be accelerated through the addition of water and elevating the temperature above 60 °C. It is well reported that this transition is thermodynamically favourable above 50 - 60 °C. The morphological changes that accompany this transition are responsible for the setting mechanism of the material.

We are investigating the use of this material as the binder in low carbon boards, as an alternative to gypsum based pasterboard, and low carbon bricks, as an alternative to clay-fired bricks. An overview of the production of these two products is given in Figure 1.

Figure 1. Brick and Board Production Schematic



Aims and Objectives

This report outlines the intermediate data collected to date on the performance of magnesium carbonate products. The aim of this deliverable is to optimise the strength of the products for appropriate applications and maximizing durability. Areas covered include:

Bricks

- Impact of curing temperature on strength
- Durability – chemical and physical effects of water

Boards

- Optimization of water:binder (w/b) for balance between product performance and rheology
- Investigating use of aggregates
- Preliminary flexural strength testing

A completed experimental plan for the remainder of this WP is also given.

Methods

Nesquehonite Production

[Redacted]

Brick Samples

Compressive Strength Testing

All testing samples were 50 x 50 x 50 mm3 mortar cubes, with the mixes described in Table 1.

Table 1. Mix designs for brick sample compressive strength testing

Testing Variable	Sample Type	w/b (w/w)	a/b (w/w)	Curing Temperature (°C)	Curing Time (days)	Testing Methods
Curing temperature	50 mm3	[Red]	[Red]	50, 60, 70	[Red]	Compressive Strength XRD

[Redacted]

A Controls Group Automatic Compression Tester was used to measure the compressive strength, with a loading rate of 0.3 MPa/s.

Durability Testing

Chemical Impact

25 x 25 x 25 mm3 cubes were prepared by combining NQ with water. These small cubes were immersed in water fully at room temperature for 1 month, 3 months, 6 months and 12 months before being removed, dried at 40 °C for 3 days. This temperature was selected for drying as it is below the critical temperature for NQ to HM transformation, preventing further transformation from introducing error to the test. The samples were then crushed using a pestle and mortar and phase analysis was completed using x-ray diffraction (XRD) using Malvern Panalytical Empyrean XRD.

Physical Impact

50 x 50 x 50 mm3 cubes were prepared using the same methodology as described in the Compressive Strength Testing section, according to the mix design given in Table 2. These cubes were dried at 40 °C to a constant mass.

Table 2 Mix designs for water absorption testing

Sample Type	w/b (w/w)	a/b (w/w)	Curing Temperature (°C)	Curing Time (days)	Testing Methods
50 mm3 cubes	[Red]	[Red]	[Red]	[Red]	XRD Compressive Strength Capillary water absorption Immersion water absorption

Capillary and immersion water absorption were both measured, following the methods giving in standards BS EN 772-11 and -21.

In the case of capillary absorption, the samples were raised such that the base of the cube and lower 5 mm of each side was submerged within the water. At specific time intervals, the cubes were removed from the water, surface dried and weighed to measure mass of water absorbed. From this, the initial rate of absorption was calculated.

For immersion water absorption, the samples were fully immersed in water. The samples were left in water until they reached a constant mass, showing they were saturated with water. This mass was recorded before samples were dried at 40 °C and cycled in and out of full immersion 10 times. Samples were taken for compressive strength testing after 1, 5 and 10 cycles.

Board Samples

[Redacted]

Results and Discussion

Bricks

Curing Temperature Optimisation

[Redacted]

Water Absorption

Capillary

Figure 5 gives the increase in mass as a function of time for brick samples during the capillary water absorption experiments. The addition of SA in the mix significantly decreases the mass of water taken up int and the rate of uptake, likely due to its hydrophobic properties.

Capillary water absorption is important to understand as it determines how much water is extracted from the mortar during construction. If the capillary adsorption is high, water can be absorbed from fresh mortars, preventing their setting. Therefore, it is important to have an initial rate of water absorption that is comparable to other commercial masonry units. These are given in Table 3.

Figure 5. Plot to show mass increase due to water absorbed as a function of immersion time

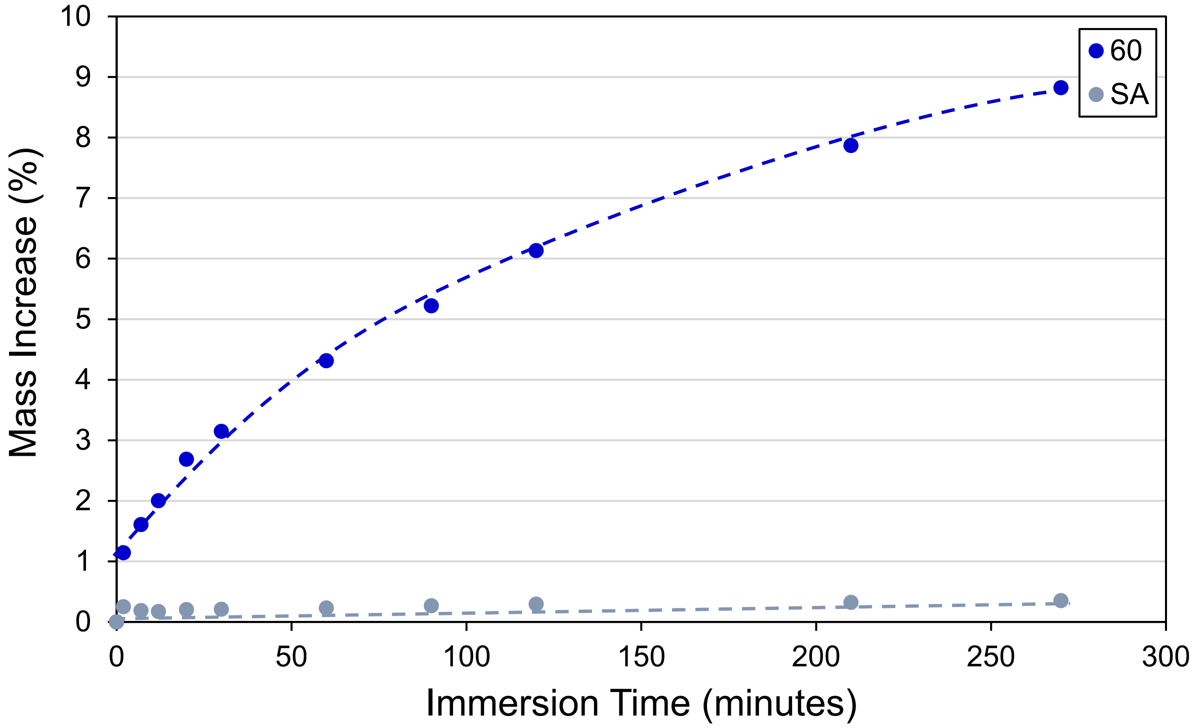


Table 3. Comparison of measured initial rate of absorption with other commercial brick products

Material	Initial Rate (kg/m2/s)
Magnesium Carbonate Brick	0.7
SA Magnesium Carbonate Brick	0.005
Ibstock Machine Moulded	0.6
Ibstock Handmade	1.9

Immersion

Figure 6 shows how the water absorption of brick samples changes with repetitive cycles of immersion, both unmodified (60) and with stearic acid (SA) included in the mix. The water absorption of the unmodified brick samples averages 16% over 10 cycles. With the addition of SA, this number is much lower, averaging 2.5%.

Figure 6 Plot to show how the immersive water absorption changes with repeat cycles and the impact of the addition of stearic acid (SA)

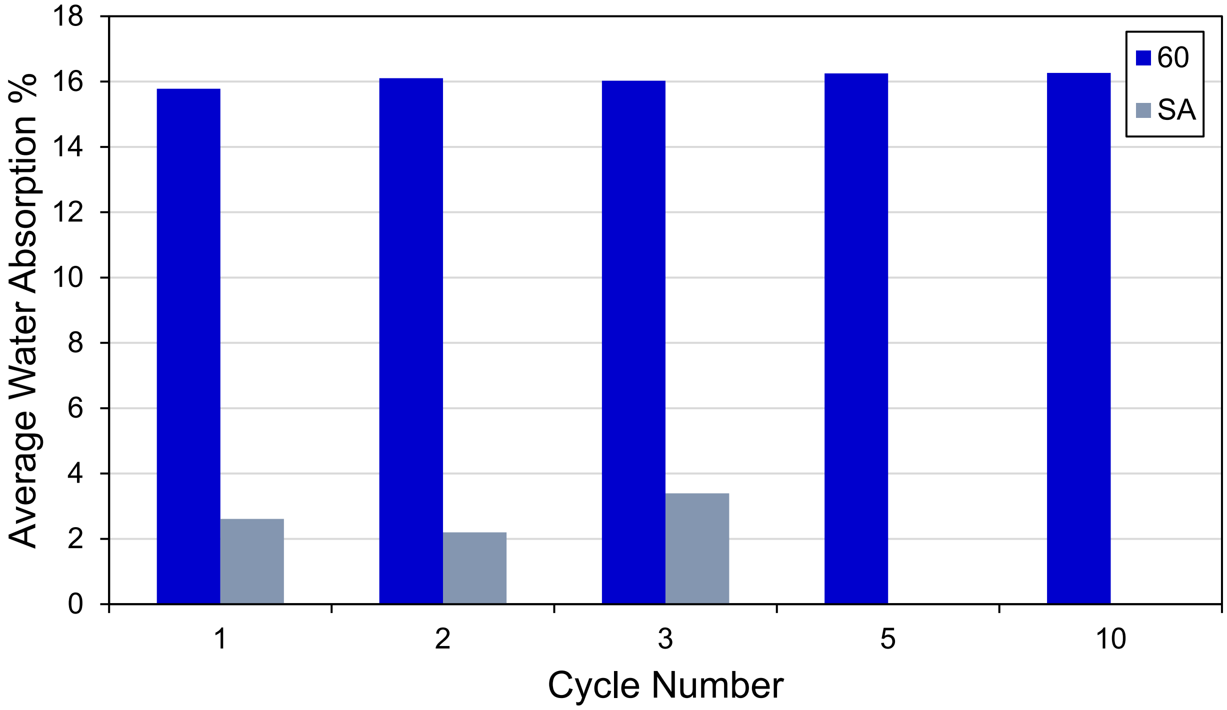


Table 4. Comparison of average water absorption with other commercial brick products

Material	Water Absorption (%)
Magnesium Carbonate Brick	16
SA Magnesium Carbonate Brick	2.4
Ibstock Machine Moulded	12
Ibstock Handmade	22

Boards

Optimisation of w/b

Current conventional board making uses a gypsum slurry, which flows on a conveyer belt between two sheets of paper before being passed through a series of ovens to dry and set. Therefore, in order for magnesium carbonate to be a direct switch for gypsum, achieving a flowable consistency is important. As observed in work completed for D4.2, the strength of magnesium carbonate decreases as the water/binder (w/b) ratio of the mix increases. Furthermore, more mixing water, results in increased shrinkage upon drying and can result in cracking. As a result, a balance must be achieved between flowability of the intermediate slurry and the viability and strength of the magnesium carbonate board.

[Redacted]

In order to understand the variable limits for further testing, board samples were made using a range of w/b and a/b to understand which were viable for further testing in D4.4. [Redacted]

Conclusions

Bricks

- No improvement on curing temperature was determined during this study. This is due to a balance of being a high enough temperature to facilitate the NQ to HM transition
- Water has no impact on the cured magnesium carbonate bricks chemically
- Magnesium carbonate brick samples have comparable initial absorption rate and overall water absorption to commercial brick samples
- Cyclic water exposure has little to no impact on the dry strengths of the magnesium carbonate brick samples
- The wet strengths of magnesium carbonate bricks are lower than the dry strengths. This must be greater than the minimum strength requirement of the use of the bricks

Boards

- Post-processing of the magnesium carbonate material was found to have beneficial effects on the final properties of curd board samples
- The composite nature of gypsum boards (paper on both sides) significantly adds to the flexural strength performance. In order to accurately compare the magnesium carbonate boards, a similar paper and adhesive must be included in future trials.

If you need a version of this document in a more accessible format, please email alt.formats@energysecurity.gov.uk. Please tell us what format you need. It will help us if you say what assistive technology you use.