IMPERIAL

Process Optimisation

CCUS Innovation 2.0
Key Knowledge Deliverable 4.2

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 a rage of pressing, extrusion and casting procedures to make magnesium carbonate construction products. Simple testing, such as compressive strength will be undertaken to identify candidate procedures to be optimised further in the remainder of the work package.

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:
industry.innovation@energysecurity.gov.uk

Contents

Introduction

Background

Phase Transition as Strengthening Mechanism

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.

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

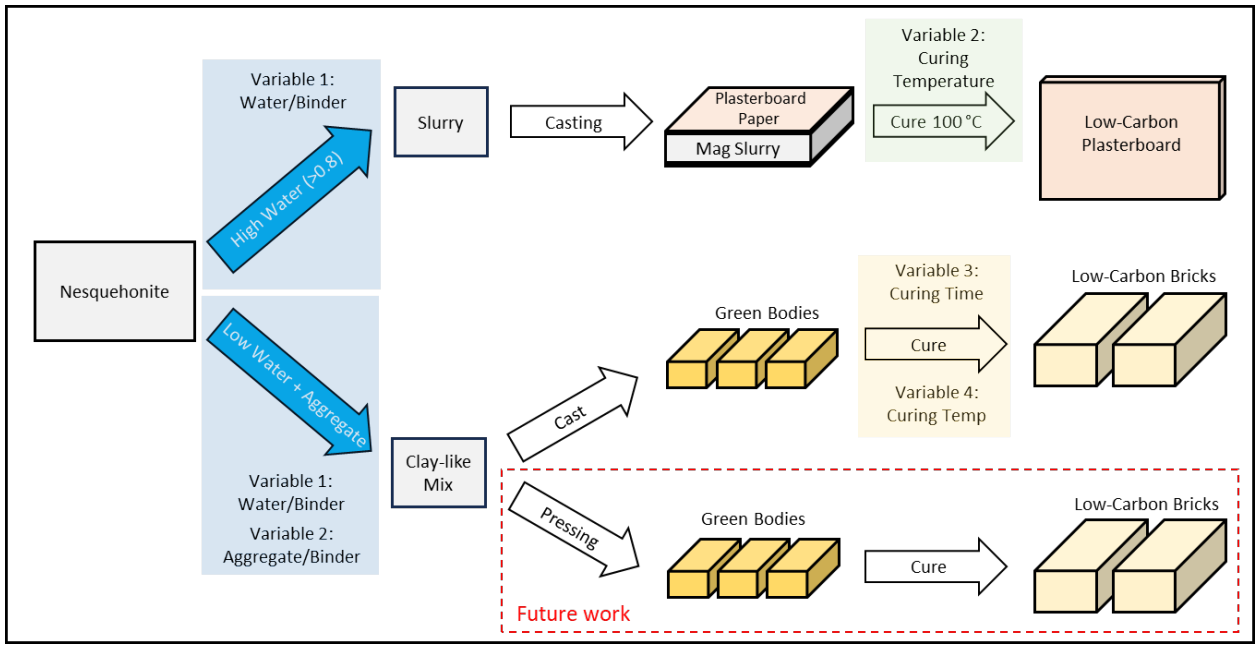
This transition can be accelerated through the addition of water and elevating the temperature above 60 °C. It is well reported that this transition is thermodynamically favourable above 50 - 60 °C [1,2].

The morphological changes that accompany this transition are responsible for the setting mechanism of the material.

Product Production

The aims of this task are to gather data to inform the optimisation of the brick and board production process. The main steps of this process are outlined in Figure 1.

Figure 1. Brick and Board Production Schematic



Aims and Objectives

The aims of this research are to optimise the magnesium carbonate product production process and maximise the performance of the resultant bricks and boards.

The main focus of this report is cast bricks. Different variables have been chosen which are imperative to the performance of these products. These variables and testing regimes are given in Table 1.

Table 1. Testing Regime of Brick/Block Production Process

Testing Variable	w/b (m/m)	a/b (m/m)	Curing Temperature (°C)	Curing Time (days)	Testing Methods
Water/binder (w/b)	0.5, 0.6, 0.7, 0.8, 0.9, 1	4	60	1	Compressive Strength XRD
Aggregate/binder (a/b)	0.6	2, 4, 6	60	1	Compressive Strength
Curing Temperature	0.6	4	40, 60, 80	1	Compressive Strength XRD SEM
Curing Time	0.6	4	60	1, 3, 7	Compressive Strength XRD

Furthermore, both pressed bricks and boards have undergone preliminary investigations to help select appropriate variables to feature in more in-depth analysis.

Methods

Nesquehonite Production

[Redacted]

Compressive Strength Testing

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

Nesquehonite and sand were added to a mixing bowl and dry mixed for 5 minutes using an electric mixer, before the appropriate amount of water was added and mixed for a further 10 minutes. This mortar mix was then added to 50 x 50 x 50 mm3 moulds and vibrated using a vibration table for 3 minutes to compact the mix. The cubes were left overnight in their moulds for water to be reabsorbed into the internal pore structure of the nesquehonite and the aggregate in order to give a thick enough consistency for them to be demoulded using an air gun. The resultant green bodies were placed in an oven at the temperature given in Table 1 for the appropriate time, to encourage nesquehonite dissolution and hydromagnesite reprecipitation. Following this, cubes are placed in ambient conditions until they reach testing age. 6 samples of each unique mix were prepared for testing.

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

X-ray Diffraction (XRD)

XRD was used to monitor the mineralogical change from nesquehonite to hydromagnesite in the samples as they set.

A representative sample of the cubes used for compressive strength testing was taken and ground in an agate pestle and mortar. The resultant powder was sieved, removing the > 75 μm fraction to isolate the magnesium carbonate paste from the aggregate.

A Malvern Panalytical Empyrean diffractometer was used over a scanning range from 5 - 80 °2θ, with Ni-filtered CuKα radiation, at a scanning rate of 0.1 °2θ s-1. Quantitative XRD can be used to identify the amount of NQ and HM present, providing information of the degree of phase transformation that has occurred.

In-Situ X-ray Diffraction

Paste samples were prepared by combining nesquehonite and water at a w/b of 0.6, and hand mixing for 5 minutes. This mixture was cast in 25 x 25 x 25 mm3 moulds and placed in an oven

at the temperature to be tested (20, 40, 60 and 80 °C). These were removed at appropriate testing times and immediately placed into the XRD for phase analysis to identify the rate at which phase transformation is taking place.

Results and Discussion

Influence of Water/Binder Ratio

The water binder ratios studied in this report are outlined in Table 1. The “standard mix design” (i.e. aggregate/binder = 4, CT = 60 °C, Ct = 1 day) was used as the set values for the control variables.

Of the proposed water/binder ration, an optimum ratio was identified providing acceptable results in key metrics such as compressive strength, workability, and durability.

Influence of Aggregate/Binder Ratio

The aggregate/binder ratio of cast samples was varied as outlined in Table 1. The “standard mix design” (i.e. water/binder = 0.6, CT = 60 °C, Ct = 1 day) was used as the set values for the control variables.

An optimal aggregate/binder ratio was determined with respect to compressive strength, workability, and durability.

Influence of Curing Temperature

The curing temperature of cast samples was varied as outlined in Table 1. The “standard mix design” (i.e. water/binder = 0.6, aggregate/binder = 4, Ct = 1 day) was used as the set values for the control variables.

An optimal curing temperature was determined with respect to compressive strength and durability. Additionally, the identified curing temperature produced samples with sufficient compressive strength after 1 day of curing to allow for manual handling.

Influence of Curing Time

The curing time of cast samples was varied as outlined in Table 1. The “standard mix design” (i.e. water/binder = 0.6, aggregate/binder = 4. CT = 60 °C) was used as the set values for the control variables.

An optimal curing time was determined with respect to compressive strength, and durability. The identified optimal curing time allowed for manual handling of cast samples immediately after curing.

Pressed Bricks

A preliminary study of pressed bricks was also conducted. 5 mix designs were decided upon with a different defining variable, as shown in Table 2.

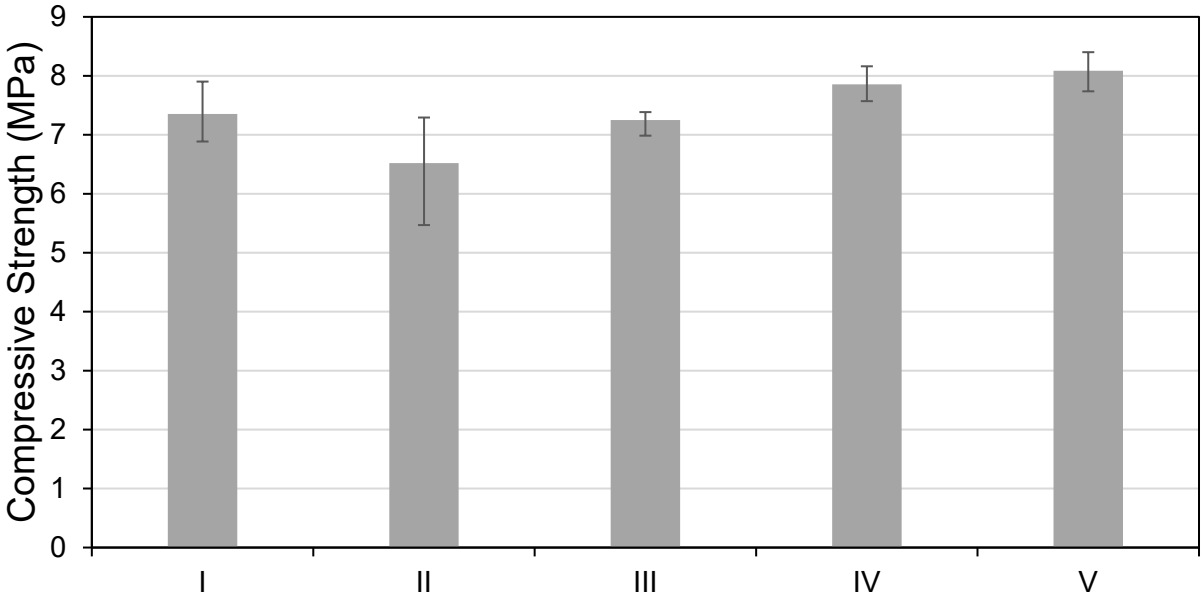
Table 2. Defining variable of the pressed brick samples

Pressed Mix	Defining Variable
I	Virgin aggregate
II	Construction waste aggregate
III	Lime
IV	Higher binder content
V	Fine aggregate only

The making of bricks via a pressed method was outsourced to Local Works Studio.

The main aim of pressing the bricks is to be able to use a reduced w/b while achieving adequate compaction, and therefore, maximising density and strength, while not compromising on degree of transformation. Unfortunately, the measurements of water absorption of the aggregates used were erroneous. Therefore, the amounts of water used in the making of the bricks was much lower than initially intended. As a result, degree of transformation for all samples was <10%. Compressive strength in all samples was approximately 7 MPa, which most likely develops due to the small amount of transformation coupled with the strength gained from the pressing pressure alone.

Figure 12. Compressive strengths of pressed brick samples


Compressive Strength (MPa)
0
1
2
3
4
5
6
7
8
9
I
II
III
IV
V

Boards

A proof of concept investigation into boards is currently being carried out as part of another EPSRC funded project. Upon the completion of this work in late May, product optimisation towards D4.3 will take place according to the work in Table 3.

Table 3. Future testing regime for boards

Variable	Testing Range	Testing Methods
Water/binder	0.8 – 1.2	Flexural Strength
Aggregate/binder	TBC	Observational Analysis Density measurements Flexural Strength
Curing Temperature	60 – 100 °C	Flexural Strength XRD SEM
Paper Grade	TBC	Flexural Strength
Types of Fiber	TBC	Flexural Strength
Fiber/Binder	TBC	Flexural Strength

Conclusions

This study allowed for the optimisation of the following parameters when manufacturing magnesium carbonate bricks.

- The ratio of water to binder (w/b)
- Elevated curing temperature
- Time spent at elevated temperature before continued curing at room temperature

The research carried out in the study shows that there is a strong relationship between degree of transformation and compressive strength of the manufactured units.

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