

**IMPERIAL**

# Dissolution Procurement

CCUS Innovation 2.0

Key Knowledge Deliverable 1.2

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 CO<sub>2</sub> gas from industrial facilities into valuable construction products. Sequestered CO<sub>2</sub> through this process is cheaper than conventional approaches that rely on purification, liquification and offshore or geological storage. The CO<sub>2</sub> 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 CO<sub>2</sub> 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**

Details of equipment procured for Dissolution stage, with invoices. Report detailing the purpose of each part. Photos / videos demonstrating equipment in location and operational.

### **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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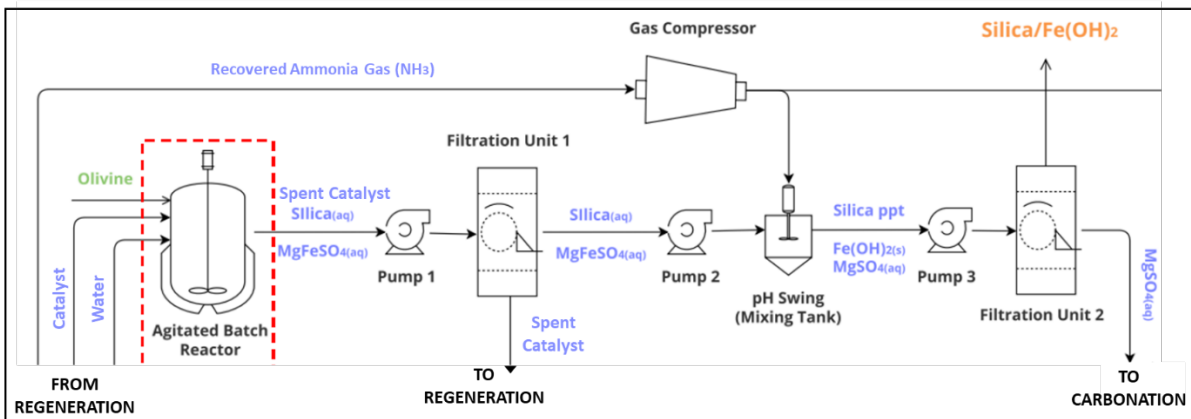
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# Introduction

The dissolution stage of the CCUS process (Figure 1) employs acidic salts to digest olivine and liberate Mg and Silica species. The activated Mg solution can contain residual iron. We have confirmed experimentally that co-precipitation of iron and silica produce an SCM with improved workability. To date, dissolution and separations experiments have been conducted at relatively small scale (< 500 ml). The following work will highlight the equipment required to operate a dissolution and separation at increased scale. Additionally, preliminary work will be conducted to ensure that the reaction products are of target purity and reaction efficiency is acceptable at these increased volumes.

**Figure 1 Dissolution and separation stage**



# Procurement

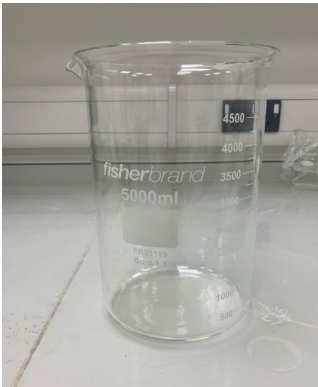
## Equipment list

Table 1 contains a list of the equipment required to perform the dissolution stage at an increased volume. The batch size achieved here is comparable to the continuous carbonation chamber batch sizes.

**Table 1 Equipment list**

Gas Item	Photo	Function	Receipt notes
5 L Glass beaker	See below	Large glass beaker in which to perform the dissolution reaction	Internally purchased from Imperial college chemical engineering store
5 L Glass Buchner flask	See below	5L side-arm flask used with a Buchner funnel to remove solids from liquids under vacuum. The Buchner funnel and flask setup are also used to remove silica and carbonate from their respective solutions.	Internally purchased from Imperial college chemical engineering store
120 mm Teflon stirrer	See below	Magnetic stirrer	n/a
Alumina crucible	See below	Large alumina crucible for regenerating the CCUS catalyst. Dissolution reactions can be done using salts or catalyst. Catalyst dissolutions at larger scales will be done in the future	Purchasing to occur in the future

**Figure 2 5L Glass beaker**



**Figure 3 5L Glass Buchner flask**



**Figure 4 120 mm Teflon stirrer**



**Figure 5 Alumina crucible**



## Rates and Reaction times

Operating the dissolution at an increased volume visually appeared to take significantly longer than smaller volume experiments. There is no chemical reason for this, however 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 typically slower than using the catalyst material. This will be explored in further detail in D 1.3.

The reaction was stopped after three and a half hours. This allows us to get a good understanding of how much longer the reaction would need without risking the reaction gelling prematurely. However, this can impact the reaction efficiency, and the properties of the silica material produced.

**Figure 6 Dissolution reaction progression**

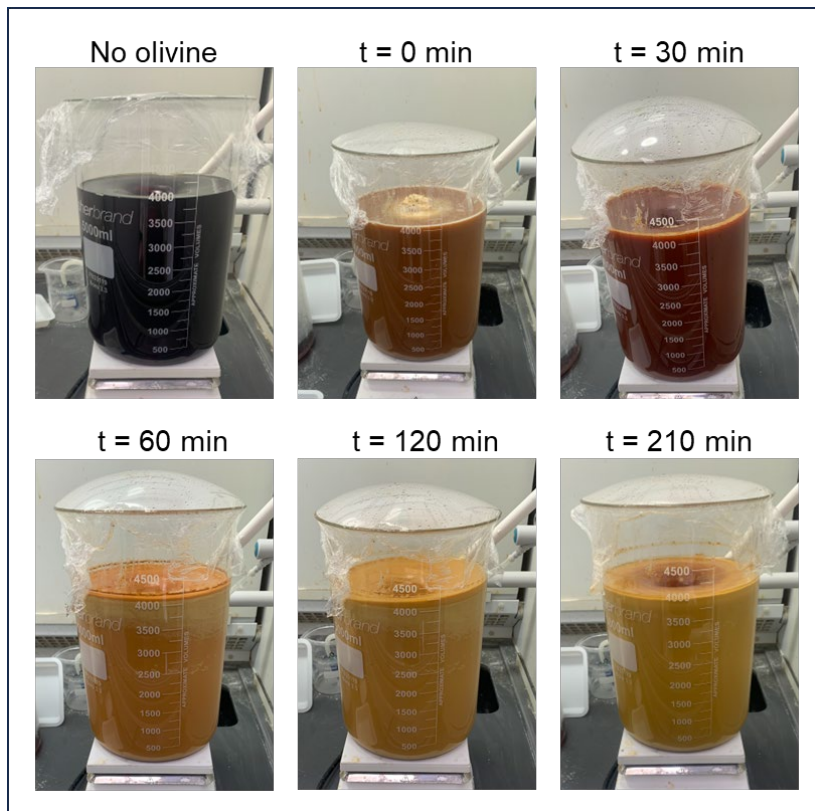


Figure 2 shows how the reaction progresses visually over time. Upon the addition of olivine the solution becomes turbid and dark orange due to rapid oxidation of iron(II). The slurry gradually becomes yellow as the reaction progresses.

After the dissolution is complete, the solid 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. The brown colour seen in figure 3 is indicative of an incomplete reaction.

**Figure 7 5L Buchner funnel filtration system**



## Reaction Efficiency

The reaction efficiency for increased volume dissolution has been experimentally determined by measuring the mass of spent catalyst produced (table 2), as well as by measuring the concentration of Mg in the magnesium sulphate solution.

**Table 2 Catalyst theoretical and percentage yields**

Expected Yield (g)	Experimental Yield (g)	Percentage Yield (%)
608	447	74

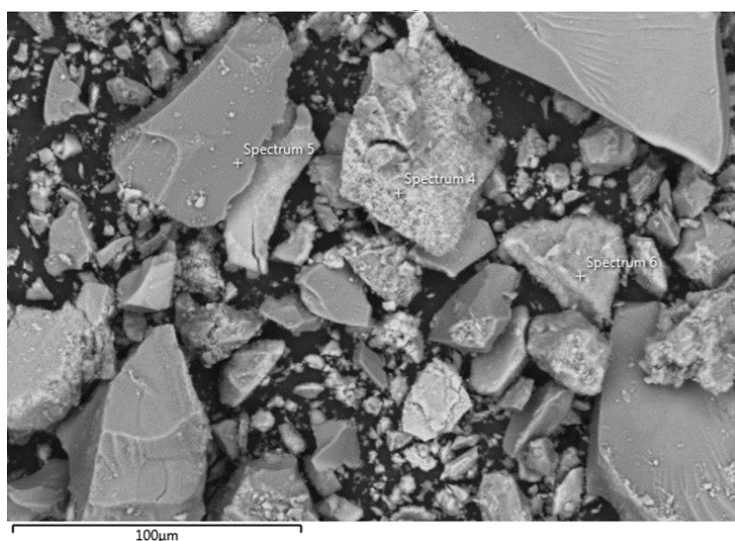
Titration data from samples taken from the end of the dissolution indicate that the concentration of Mg in the solution is 0.68 moles/L. The theoretical maximum concentration that the magnesium sulphate solution can reach is 0.95 moles/L. The effective completion of the reaction is calculated to be 72 %. This shows close alignment with the spent catalyst mass.

## Product Characterisation

As a result of the reaction not being completed, the produced silica contains significant quantities of iron hydroxide species. To confirm this, SEM images were taken, and EDS analysis was performed on the dried silica sample.

Figure 4 shows the SEM of the silica, as well as the points analysed by EDS, the oxide compositions are described in table 3.

**Figure 8 SEM image of silica produced from large dissolution.**



**Table 3 Oxide composition of silica (EDS)**

Oxide	Spectrum 4	Spectrum 5	Spectrum 6
SiO <sub>2</sub>	[redacted]		
Fe <sub>2</sub> O <sub>3</sub>			
SO <sub>3</sub>			
MgO			
NaO			
K <sub>2</sub> O			
Al <sub>2</sub> O <sub>3</sub>			

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