IMPERIAL

Flue Gas Recovery & Testing 1

CCUS Innovation 2.0

Key Knowledge Deliverable 1.1

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 testing of first flue gas(es). Report to include: source, composition, capture efficiency, reaction time, characterisation of magnesium carbonate product and residual solution.

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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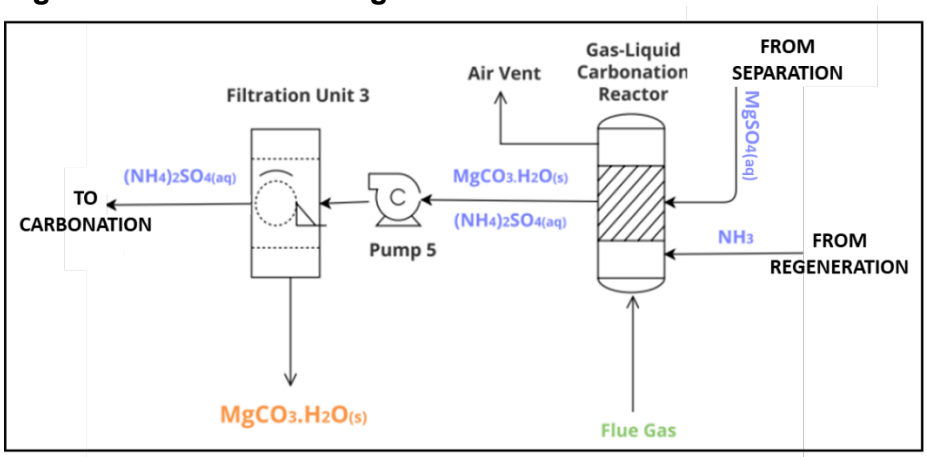
Contents

Introduction

The carbonation stage of this project's carbon capture and storage (CCUS) process (Figure 1) is a liquid-gas reaction between activated Mg and the CO2 present in flue gas streams. The activated Mg solution can contain residual iron. We have confirmed experimentally that the presence of iron does not have a negative impact on the rate of CO2 sequestration. The following work will show that the presence of iron in the solution does not impact the type of magnesium carbonate (MgCO3) formed during the mineralisation. This means that any MgCO3 product that CCUS has developed will exhibit the same properties regardless of the iron content in the starting magnesium silicate mineral.

Figure 1 Carbonation Stage



Experimental

Source composition

Four gas compositions have been trialled at lab-scale; 1) Compressed air, 2) Pure CO2 stream, 3) Simulated flue gas one, and 4) Simulated flu gas two. Table 1 outlines the compositions of the gases. Besides the compressed air, all gases were purchased from BOC.

Table 1 Gas compositions

Gas Type	CO2	CO	N2O	SO4	O2	AR	N2
Air	0.04	-	-	-	20	0.9	78
CO2	>99.9	-	-	-	-	-	-
Sim Gas 1	15	590 ppm	105 ppm	-	10	-	~75
Sim Gas 2	-	-	-	5 ppm	-	-	~99

Rates and Reaction times

The rate at which CO2 mineralises is determined by how quickly CO2 dissolves into the activated Mg solution. In practice, this manifests itself in two ways; either the concentration of the CO2 could be increased, or the flowrate of the gas stream can be increased. Either of these levers, provided there is sufficient activated Mg in solution, and the contact area is large enough, will increase the rate of sequestration.

We have investigated carbonation at three different concentrations of CO2: 0.4, 15 and 100 %. Attempts using 0.4 % (400 ppm, atmospheric concentration) have not yielded any precipitate after 10 days of carbonation. Direct air capture using activated magnesium may be possible but would require unreasonably large spaces to make the carbonation rate practical. The research focus has instead been on pure CO2 and simulated flue gases.

We have investigated the rate of carbon mineralisation across a range of different magnesium concentrations. The concentrations studied were 0.1, 0.2, 0.3, 0.4 and 0.5 mol/L. The “pure CO2 stream” was chosen for these studies to minimise external impacts. Figure 2 shows the relationship between Rmax the maximum rate of carbonation in each experiment (in kg/m3/hr), and the magnesium concentration (in mol/L).

Figure 2 Impact of magnesium concentration on Rmax

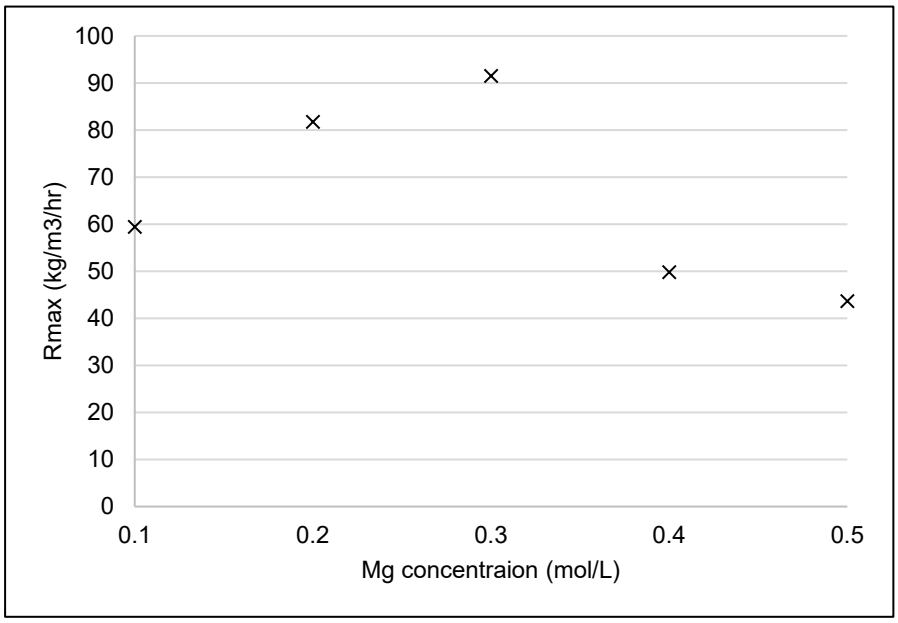
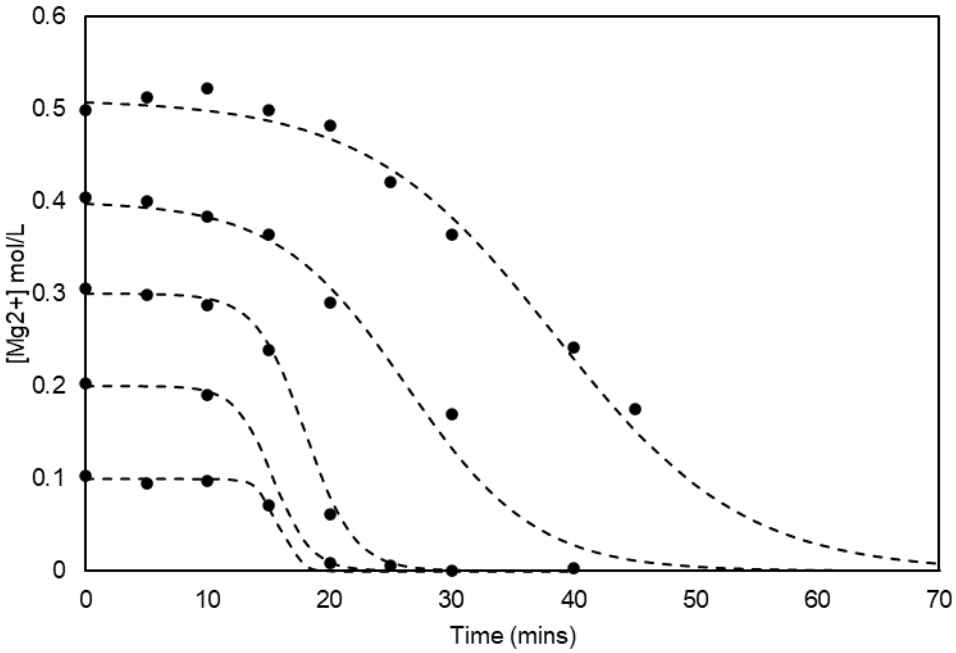


Figure 2 shows that increasing the amount of magnesium in the solution increases the rate. However, at higher concentrations the amount of precipitate thickens the slurry and reduces efficiency, reducing carbonation rates. Initial rate measurements of 2.0 M Mg solutions show an Rmax of 158 kg/m3/hr. For context, this would be fifty times the rate of sequestration compared to direct air capture, for a given volume.

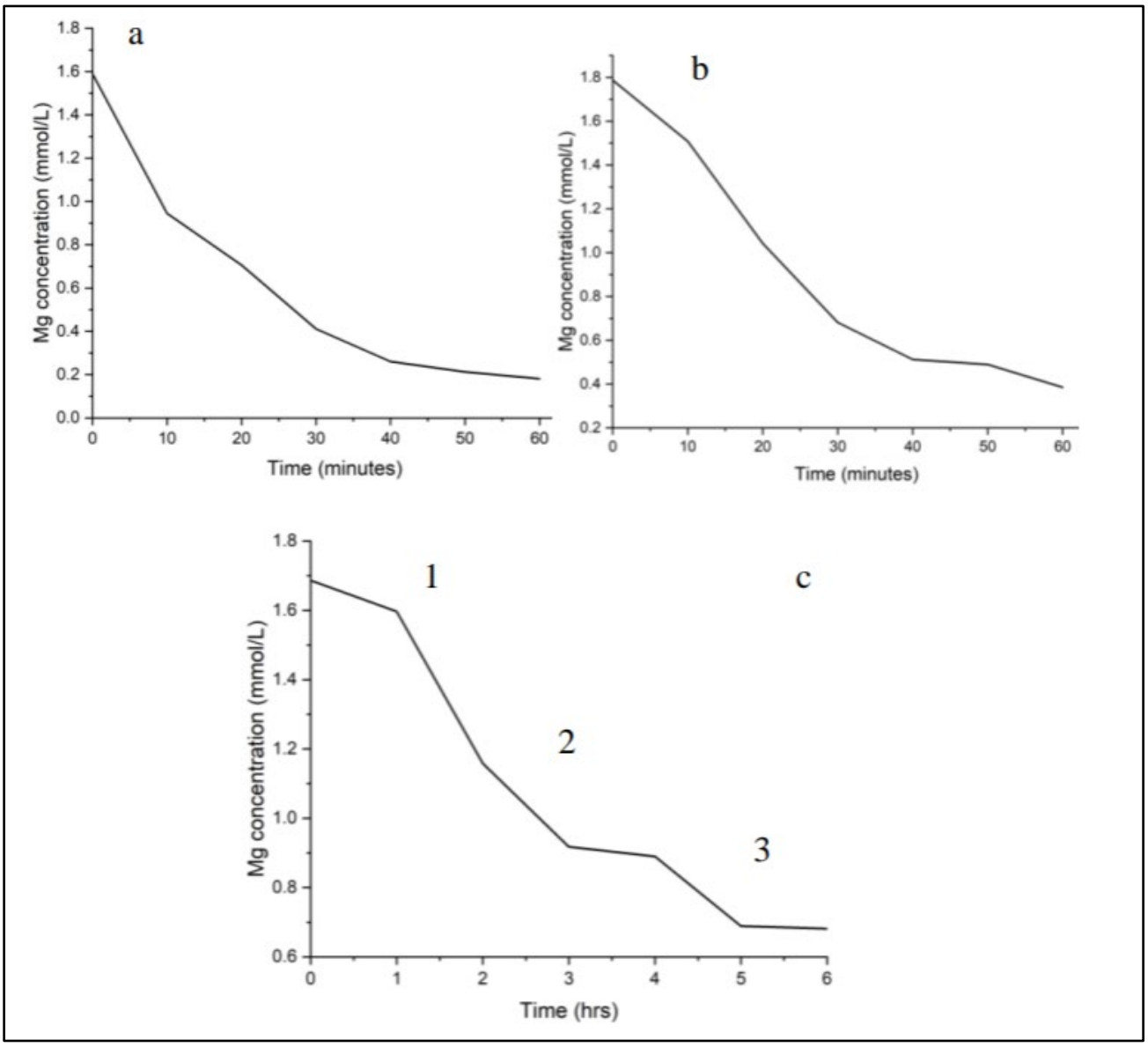
Figure 3 Magnesium concentration in carbonation liquid v time (100 % CO2, 2 L/min, initial concentration dictated by [Mg2+] at t = 0



The Rmax values are the highest value in the derivatives of graphs such as those in Figure 3. Figure 3 shows how the whole reaction progresses over time. We have observed that the

reaction occurs in three stages, and that these stages are more visible at lower CO2 concentrations. These stages are 1) incubation, the solution is undersaturated with CO2 and no precipitate is forming, 2) steady-state, the precipitation is occurring at the maximum rate for that system, and 3) trailing, where precipitation rate decreases with decreasing magnesium content. These stages are visible in Figure 4(c), when using simulated flue gas.

Figure 4 Magnesium concentration in carbonation liquid v time



Capture Efficiency

Capture efficiency (μCO2) is the amount of CO2 mineralised by the aqueous system as a proportion of the total CO2 that passes through the system.

$$\mu_{CO2} = \frac{Rmax}{\rho CO2 \times F \times P}$$

Where pCO2 is the partial pressure of CO2 in the gas, F is the gas flow in L/min, and P is the pressure the gas is under.

In the case of “pure CO2,” we will take the Rmax value of 158 kg/m3/hr. The total amount of CO2 is passing through the aqueous system is 440 kg/m3/hr. This gives a capture efficiency on a single pass of 35%.

In the case of simulated flue gases, the Rmax value is 22 kg/m3/hr, and the total amount of CO is 66 kg/m3/hr. This gives a single pass capture efficiency of 33%.

The observation that the capture efficiency of this system seems unaffected by the concentration of the CO2 present in the gases 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.

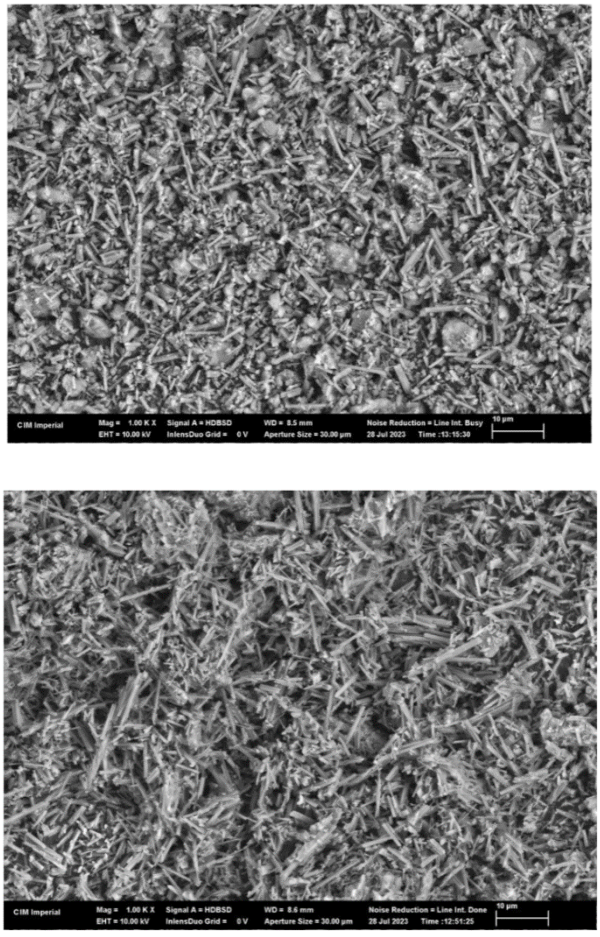
Product Characterisation

Electron microscopy

The magnesium carbonate produced has been characterised by SEM, X-Ray diffraction, and PSD. The gas composition could have impacted the chemical phases present in the solid sample, or the mechanism or precipitation.

Figure 5 shows the SEM images of the carbonate product with CO2 captured from a “pure CO2 stream” (left) and from a simulated flue gas (right). As the images show, there is no difference in crystal structure across the two samples. This indicated that the chemical species present the flue gas does not have a direct impact on the morphology of the carbonate produces.

Figure 5 SEM image of one mol/L Mg solution carbonated with “pure CO2” (top) and simulated flue gas 1 (bottom)



Particle size analysis

Particle size analysis, determined by laser diffraction, was used to further investigate how particle morphology is impacted by gas composition and CO2 concentration. Table 2 contains the size distribution of the magnesium carbonate particles present. The carbonate particles produced from simulated flue gas are consistently larger, owing to the longer reaction time overall. Slower grown crystals tend to be larger, and a long residence time allows for mechanisms such as Ostwald ripening to coarsen the particles. These larger crystals can also be seen in Figure 5.

Table 2: PSD of magnesium carbonate produced by pure CO2 and simulated flue gas.

Size Fraction	Pure CO2	Sim Gas 1
D10 (um)	3.7	4.1
D50 (um)	8.6	16.7
D90 (um)	22.2	35.4

X-ray diffraction

X-ray diffraction analysis provides insight into any impurities that may be present within the carbonate product. Figure 6 shows that despite high concentration of SOx and NOx in the simulated flue gas, the only insoluble phase present in the carbonate product is magnesium carbonate. Magnesium sulphate salts are present due to partially incomplete reactions and ammonium sulphate be precipitate out of solution upon drying. However, both are easily removed by washing the sample if required. Figure 7 shows the diffractogram of an unwashed magnesium carbonate sample produced with simulated flue gas one.

Additionally, the XRD analysis shows that despite altering the gas composition, the polymorph of magnesium carbonate is the same in both cases, nesquehonite. The type of carbonate formed is more greatly influenced by factors such as pH or temperature, both of which are more easily controllable than the composition of flue gases.

Figure 6 XRD of magnesium carbonate product from pure CO2 and simulated flue gas

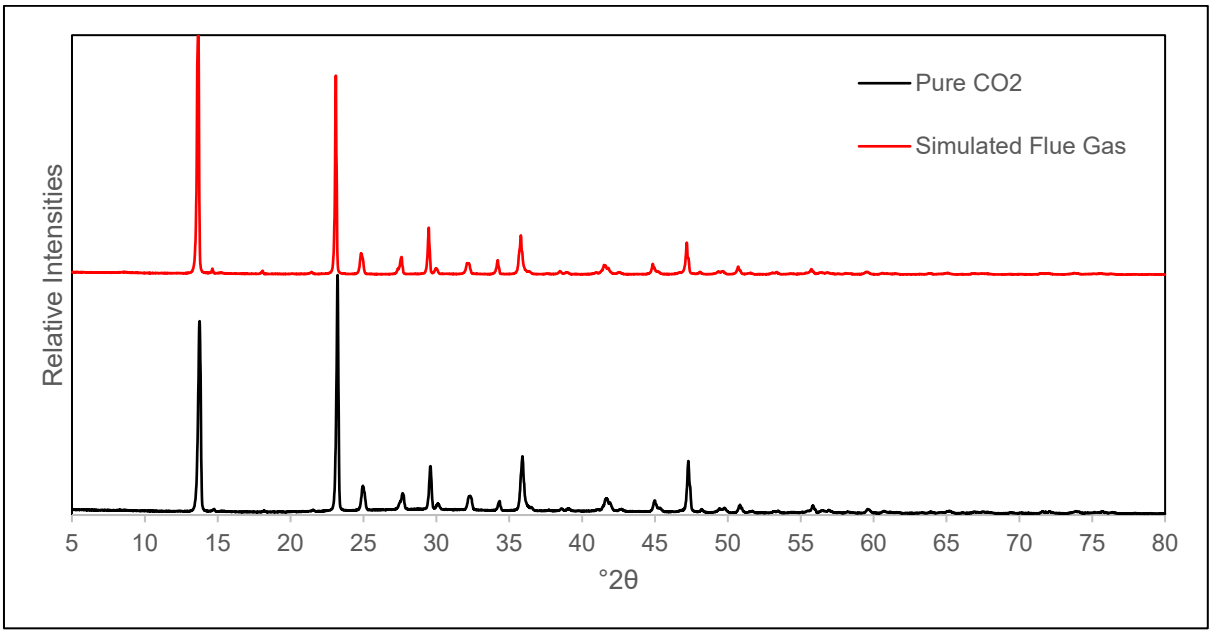
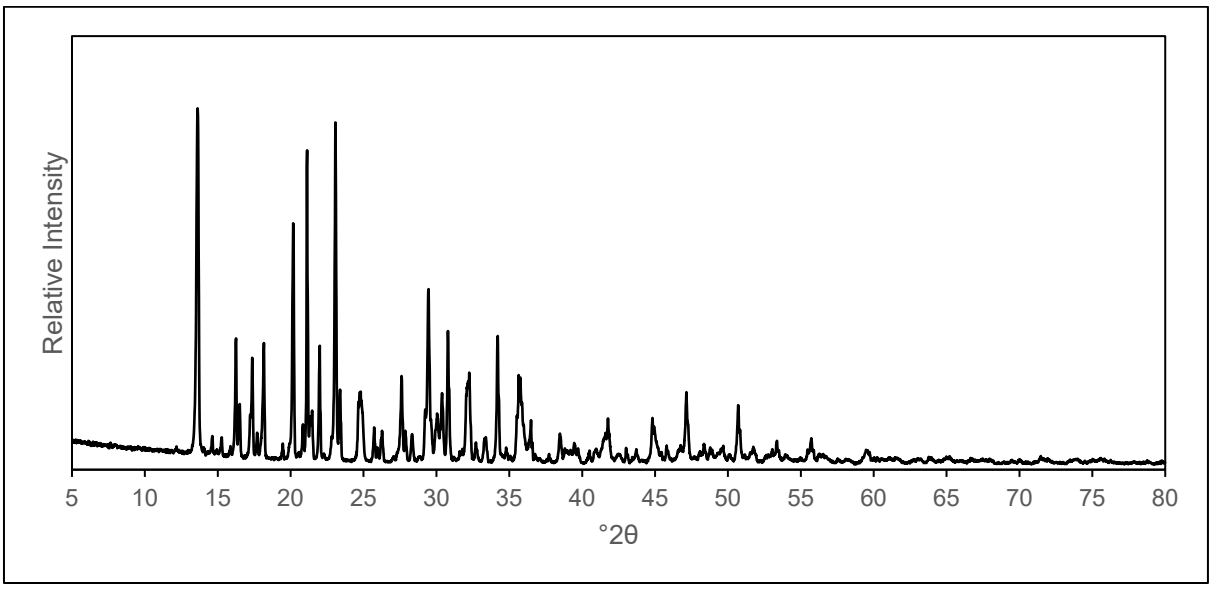


Figure 7 XRD of unwashed magnesium carbonate product from simulated flue gas



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