Chapter

Testing and Validating Instruments for Feedstocks of Mineral Carbonation

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Abstract

Different feedstocks Dunite, Olivine and Lizardite are examined in this research using various measuring techniques such as TGA-MS, XRD and Quantitative XRD and EDS. Quantitative XRD results matched with TGA-MS results. Malvern Mastersizer, EDS and QXRD results also showed a good match regarding the individuality of results which are shown graphically. TGA-MS calibration curves example is provided. Matching the results of different measuring techniques is a key to fundamental research. Comparison of the reactivity of dunite, soaked dunite, heat-activated dunite and lizardite and raw dunite soaked has been performed. TGA-MS and QXRD results match each other. Malvern Mastersizer, EDS and QXRD results match with their individual results indicating the instrument’s reliability. Semi-Quantitative XRD results authenticity is EXCELLENT. TGA-MS results match with QXRD is excellent. Mineral carbonation converts CO2 into stable mineral carbonates. This research explores the utilisation of serpentinised dunite (which is comprised of 61% lizardite) as a potential feedstock for mineral carbonation. Heat activation, ex-situ regrinding and concurrent grinding techniques were employed to enhance the reaction rate and yield, and to provide information on the carbonation reaction mechanism. Silica-rich layers that appeared during reference experiments were disrupted using concurrent grinding and significantly higher magnesite yields and Mg extractions were obtained.

Keywords: CCS (carbon capture and storage), CCSU (carbon capture, storage and utilisation), mineral carbonation (MC), carbon capture (CC), materials science

1. Introduction

Greenhouse gases especially CO2 concentration in the atmosphere has increased to a level of 419 ppm compared to a value of 280 ppm from the preindustrial revolution (1975) [1]. Reduction in greenhouse gases is a need of time. Significant research has been published regarding mineral carbonation [2–15], geological carbon dioxide storage, oceanic storage [3], carbon dioxide conversion into chemicals, carbon dioxide fixation in polymers and carbon dioxide conversion into Urea [16, 17]. Mineral carbonation is one of the forefront technologies recently proposed. Although various publications have been done in this field [2], the basic need of time is to foresee how
the research efforts need to be oriented or centred on that technology [3, 4, 9–11, 13, 14, 18–20]. This article will indicate some directions for the utilisation of different feedstocks for CO₂ utilisation and fixation. Geological storage poses a threat to nearby occupants as there were thousands of killings in the Crater Lake incident. ALOHA software can be used for the estimation of such leakages if CO₂ is to be stored in geological formations. CO₂ can cause asphyxiation; hence, safety measures are at most necessity. ALOHA can estimate CO₂ vapours travelling distances and how far this gas can travel and how much concentration will be at a specified point. Oceanic storage pose threat to aquatic life and is expected to disrupt the ecosystem seriously. Increased concentration of CO₂ in oceans [21] will reduce the pH of the oceanic water, rivers or canals making them undrinkable. Seawater is used in various industries. Reduced pH will cause serious corrosion issues and may result in materials damage and or stress corrosion cracking.

Greenhouse gases are uncontrollable. Each greenhouse gas concentration increases day by day. CO₂ is recently converted to jet fuel using sunlight by Adele Peters from Fast Company [22]. Researches are not giving up. However, the Antarctic lake has disappeared in just 3 days [23]. More efforts need to be initiated. Extremism in climate shattering weather patterns is expected right now [23]. Europe has seen extreme flooding in 2021. Pakistan has seen extreme summers like what the Middle East has seen shooting of temperatures. Catalysts have been discovered to convert CO₂ into fuel [24]. A single reason why CO₂ is not controlled is that industries emit more than capture. Adam Vughan has indicated that atmosphere warming could not have been kept below 1.5°C [25]. Alas, more seriousness is required. No negative emissions drama. The cement industry is also one of the largest CO₂ emitting industries. Novel modifications are proposed to overcome this threat [26]. Coal-burning emissions and their environmental effects are also highlighted [27].

2. Analytical instruments

Dunite, different varieties of olivine and lizardite are used in this research. Proper functioning of analytical instruments is a fundamental to perform the highest level of research. Fundamental instruments operation and working is described here.

2.1 TGA-MS analysis

Thermogravimetric analysis (TGA) measures the change in mass over time as the sample is heated. These measurements provide compositions of different feeds or carbonated products. TGA is suitable to characterise different materials that display mass loss or gain due to thermal decomposition and thus enable an estimate of magnesite yields of the carbonated products to be obtained. Feed materials or carbonated products were heated in TGA (Figure 1) from 25 to 1000°C and mass losses due to decomposition of different phases present are identified. To identify the evolving gases generated during heating, the TGA-DSC (Setsys Evolution 1200) was coupled with a mass spectrometer (Thermostar Quadrupole). The initial loss of mass observed between 25 and 280°C corresponds to physically bound moisture present in the sample, while the second mass loss from 280 to 430°C corresponds to brucite decomposition, while the third major mass loss in the range of 430–830°C corresponds to lizardite decomposition (Figure 2).
Lizardite decomposes over the same temperature range (300–600°C) as the magnesite [6] and this can introduce a systematic error in magnesite yield estimation (leading to an over-estimation of the magnesite yield) unless the mass loss in this period can be quantitatively attributed to loss of H$_2$O or CO$_2$ from the sample. To distinguish between these species, the ion current from the m/z = 44 ion (CO$_2^+$) from mass spectrometer was calibrated using sodium bicarbonate samples and a calibration curve for CO$_2$ concentration was obtained, which is used to quantify CO$_2$ mass loss (distinguishing CO$_2$ production from the loss of water vapour, which occurs simultaneously) and thus render more accurate estimation of the magnesite yield. CO$_2$ peak areas were calculated using the mass spectrometer data and CO$_2$ mass response is estimated based on the reaction (2NaHCO$_3$ $\rightarrow$ Na$_2$CO$_3$ + CO$_2$ + H$_2$O). The relationship between peak areas and CO$_2$ mass loss was linear.

For carbonated samples, the CO$_2$ peak areas were estimated using MS data and then these peak areas are used to determine CO$_2$ mass loss applying the CO$_2$
calibration curve. This CO₂ mass loss was used in an equation to calculate magnesite yield. The equation is based on the Gadikota formula [28].

Three TGA runs (Table 1) were completed with calcium oxalate hydrate (99% pure) to calibrate the TGA response. Theoretical and measured mass loss shows good agreement (Table 1).

### 3. XRD analysis and QXRD (semi-quantitative XRD)

XRD analysis is useful in the determination of crystalline phases that exists in a powdered sample [30]. Each phase has a specific, identifiable x-ray diffraction pattern, which is used to determine different phases present in the sample. X-rays are generated from the emission of high energy electrons from hot tungsten elements, which are bombarded on a copper metal target. This bombardment causes an electron emission from target atoms, thus generating an electron vacancy which is filled by an electron from higher energy orbitals and this transition generates x-rays. Filtration of these x-rays is performed to get monochromatic radiation which is bombarded on the sample being analysed. Bragg’s equation is the main law used in XRD diffraction pattern analysis [31].

\[
n\lambda = 2d\sin\Theta
\]

\(n\lambda = 2d\sin\Theta \tag{1}\)

\(\lambda = \) wavelength of x-rays, \(n = \) integer, \(d = \) plane spacing, \(\Theta = \) Bragg’s diffraction angle.

To derive Bragg’s law, consider two x-rays (A and D) impinging on the atom B and E of a crystal and the angle of incident and angle of reflectance are equal as shown in Figure 3. Incident waves A and D are in phase with each other although wave D has to travel an extra distance of GE + EH to remain in the same phase as wave A. This extra

![Figure 3. Schematic of X-ray diffraction (left), Bragg–Brentano geometry (right).](image-url)
distance must have been an integral \((n)\) multiple of wavelength \((\lambda)\). The length \(GE\) and \(EH\) are equal and \(GE\) equals to \(d \sin \theta\). Bragg–Brentano design is the most commonly used instrument geometry for high-resolution powder diffraction. The incident beam through a number of slits diverges towards the sample, the diffracted signal from the sample again converges through a number of slits towards the detector. A \(\Theta/2\Theta\) rotation is employed to keep incident and diffracted wave paths in symmetry. During sample scanning, the sample rotates by \(\Theta\) while the detector is rotated by \(2\Theta\) with each step [32].

In practice, finely ground feed materials and carbonated samples (up to 100 \(\mu\)m size particles) were put in the instrument holder for analysis. Samples were finely grounded in order to avoid intensity fluctuations and preferred orientation. XRD analyses were performed using Philips X’Pert Pro multipurpose diffractometer with Cu radiation and \(2\theta\) from 5 to 90° or 11 to 31° depending upon the sample being analysed. Collection time used was 1 s with a step size of 0.02°. The patterns from XRD were matched with the International Centre for Diffraction Data ® (ICCD) using X’Pert Highscore® in order to identify crystalline phases. A typical x-ray diffraction pattern for feed dunite is shown in Figure 4. Phases identified are lizardite, olivine, brucite and magnetite.

If the reference intensity ratio (RIR) of an analytical phase \(i\) (such as silicon) is known, then its concentration can be calculated by doping the original sample with the analytical phase. This can be done by the addition of a known amount of standard (silicon) of which the RIR is known. For semiquantitative method details please see below.

After obtaining the diffraction pattern of the doped sample, the concentration \(C_i\) in the original sample is calculated as follows:

\[
C_i = A_x \times \left(\frac{I_i}{I_x}\right) \times \left(\frac{RIR_x}{RIR_i}\right)
\]

\(C_i = \) concentration of given phase \(i\) in the original sample  
\(A_x = \) known amount of standard (silicon) added to the original sample

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**Figure 4.**  
Typical x-ray diffraction pattern for dunite. L, Lizardite; O, olivine; B, Brucite; M, magnetite.
Mineralogy

\[ I_i, I_x = \text{intensities (peak areas) of phases } i \text{ and } x \text{ in the doped sample} \]
\[ \text{RIR}_i, \text{RIR}_x = \text{reference intensity ratio values of } i \text{ and } x \text{ respectively} \]

The procedure is described below in detail (for calculation detail see Table 2)

1. Add known amount of silicon (usually 20 wt/wt%) in the sample and scan it with XRD

2. Identify phases present in a given XRD pattern

3. Determine the area under the main peak of each phase. The peak should not overlap other peaks. To calculate the area under the main peak, first, calculate slope (M) and then intercept (C). Then calculate derivative (\( Y' \)) and use the given formula to calculate the area under the peak (please refer to Table 2). Use these peak areas in Eq. (2).

4. The RIR value is found from the Highscore® database

5. The concentration \( C_i \) of each phase is estimated using Eq. (2)

3.1 ICP-OES (inductively coupled plasma: Optical emission spectrometry)

The elemental composition of solid and liquid samples can be determined using ICP-OES. ICP-OES consists of two major components; the torch and optical spectrometer. The torch comprises quartz tubes [33]. To produce plasma, argon gas is normally used, which passes through the tubes around the induction coil. The argon gas is “ignited” by the Telsa unit and the ionisation process (plasma formation) is initiated. The ionisation of argon gas occurs at this stage. A plasma having approximately 7000 K temperature is generated because of collisions between neutral argon atoms and charged particles [34]. Using a peristaltic pump, an aqueous sample is continuously supplied to the nebuliser where it changes to mist and moves to the plasma envelope. The introduced sample interacts with electrons and ions in the plasma and is converted into charged ions. This causes the decomposition of different molecules into respective atoms that lose electrons to induce the emission of radiation of distinctive wavelengths of elements present inside the sample. The optical spectrometer separates these wavelengths into component wavelengths. Intensities are compared with the intensities of standard solutions of known element compositions.
and elements concentrations are computed based on the calibration curves. ICP-OES set-up and microwave digestion system is shown in Figure 5.

Solid samples used in the present investigation were first digested in acidic solution. Dunite sample (0.1 g) was digested in a microwave oven (Figure 5) using a mixture of 4.5 mL HNO₃ (65%), 4.5 mL HCl (37%) and 3 mL HBF₄ (tetrafluoroboric acid, 50%). Thulium (50 μL) was added as a tracking element. The volume of this mixture was increased to 20 mL by the addition of 2% nitric acid prior to its digestion in the microwave. Digestion was not required for supernatant solution samples and they are diluted using 2% nitric acid to the required level (50%/100% dilution) prior to their analysis by ICP-OES (Varian, Australia). The typical curve for ICP-OES is shown in Figure 6. Mg concentration drop with the passage of time due to magnesite precipitation. Si concentration increases during the first hour but then it stays constant, which is due to simultaneous silicon leaching from dunite and its precipitation in the form of silica.

Figure 5.
Photo of the ICP-OES set-up and microwave digestion system. A, autosampler with standards and sample tube holders; B, plasma chamber; C, gases exhaust; D, computer for analysis output; E, argon gas cylinders; F, chiller; G, microwave digestion system.

Figure 6.
Typical curve for ICP-OES. The graph represents Mg and Si concentrations variation with time for supernatant solution of sub 75 μm heat-activated dunite carbonated sample. Carbonation reaction was performed with 15% solids slurry at 185°C, 130 bar pressure and using 0.64 M NaHCO₃.
3.1.1 Scanning Electron microscope (SEM)/energy dispersive scattering (EDS)

Morphology, surface topography and elemental compositions of feed materials and carbonated products were determined using SEM (Zeiss Sigma VP FESEM) and EDS (Bruker). SEM scans a fine electron beam over the material being analysed and uses different detectors to reconstruct the image from signals produced from the sample [35]. SEM consists of different parts, e.g., microscope column which also includes electron gun and electron beam travels in this column; the computer that drives the microscope; ancillary equipment which analyses the composition. SEM can magnify objects from 10 times to 300,000 times. Scanning from an electron microscope can be compared with a person having a torch and looking for objects on the wall. As a person builds an image in his/her memory, SEM works in the same way and uses a fine electron beam instead of the torch to build an image.

EDS is a technique that provides information about the chemical composition of the sample. For EDS, an electron beam is focussed on the sample during SEM analysis and these electrons interact with the atoms. X-rays are produced from these interactions and an energy dispersive detector detects these x-rays and displays a signal in the form of spectrum, histogram or intensity versus x-ray energy. This makes it possible to identify elements present in the sample.

Sample preparation is important for SEM. Samples are gold (imaging) or carbon (EDS) coated prior to their analysis. Gold coating provides a thin layer to the samples and samples were coated four times at a 90° angle and fifth time from the top. A typical SEM micrograph and EDS spectrum of dunite feed sample are shown in Figure 7. SEM shows an image of the dunite feed and the EDS spectrum indicates intensities of the elements present in the sample. Polished resin blocks were used to study the silica-rich layers. Polished resin blocks were prepared using feed material, carbonated products and resin. Photo of polished resin blocks and sample holders is shown in Figure 8. The polished resin block samples preparation procedure is given in appendix 3D.

3.1.2 Transmission Electron microscope (TEM)

TEM is useful to study the structure, properties and compositions of different mineral powders, especially in the submicron range. Mineral particles should have
been in 50–100 nanometres size to be properly analysed by TEM. Electrons transmis-
sion through the mineral particles enables detailed analysis of the particle features
especially its crystal structure, orientation and chemical composition. In the present
study, TEM was used to identify shell (silica-rich layers) and core part of the reacted
mineral particles and study the corresponding elemental compositions and structure.

To prepare samples for TEM (JEOL 2100 TEM) analysis, 10 mg of sample powder
was added to the pestle and mortar. Ethanol (4 ml) was mixed with the sample
powder and contents were ground for 3 min. Ground sample was moved into a 5 ml
plastic vile and sonicated for 20 min. Using pipette 1–2 drops were dropped on the
TEM grid (200 mesh Cu, ProSciTech) and air-dried overnight prior to TEM analysis.

3.1.3 Fourier-transform infrared (FTIR) spectroscopy

FTIR is useful to acquire an infrared spectrum of a solid sample either feed or
product. In FTIR spectroscopy, most molecules absorb light in the infra-red region of the
electromagnetic spectrum and this absorption corresponds to the specific bonds present
in the molecule. Measurement usually is in wave numbers typically over the range
4000–400 cm\(^{-1}\). When the sample being analysed is exposed to radiation, some portion
of the radiation is absorbed while other is transmitted. The block diagram of the FTIR
spectrometer is shown in Figure 9. Dunite and carbonated products were analysed by
FTIR (Bruker, Tensor 37 Spectrometer). Samples for FTIR spectroscopy were prepared
using 99% KBr and 1% sample. Samples were thoroughly mixed and ground before
making pellets. These pellets were placed in an oven under a vacuum at 150°C for
24 hours before analysis. Typical FTIR spectra of raw dunite are provided in Figure 10.

3.1.4 Malvern Mastersizer

Particle size distribution (PSD) of feed materials and carbonated products were
determined using a particle size analyser (Mastersizer 2000, Malvern Instruments)
(Figure 11). For particle size measurement, it is important how particles scatter and
absorb light. Initially, the Fraunhofer model [36] was used to predict scattering
patterns when a solid disc of particles is passed through the laser beam. This
model failed to describe exact scattering as very few particles are disc-shaped. Mie
theory is currently used which is able to predict the light scattering behaviour of all
materials. Each size of particle has a specific scattering pattern. The particle size
analyser uses the above-mentioned theories and works backwards to calculate particle
size from the captured scattering pattern. Basic laser diffraction system is shown in Figure 12.

Three procedures are used for any particle size measurement. First, a sample is prepared and dispersed in a dispersion unit in proper concentration followed by its delivery to the optical bench. Second, a scattering pattern is captured from this sample which is also called “measurement” and is done by the optical bench. Third, raw data from measurement is analysed by instrument software to provide the PSD. Finely ground samples (≤20 μm) were preferably analysed in wet solution form. These samples tend to agglomerate during drying even if dried under vacuum and at low...
temperatures (70°C). For these samples and heat-activated samples, a built-in ultrasonic system in the Malvern mastersizer was used to break any agglomerates present. A typical PSD from the Malvern mastersizer is shown in Figure 13. Size classes are represented on the x-axis in μm and the volume density of particles is represented in percentage on the y-axis. The percentiles, d10, d50 and d90 are shown in Table 3. D10 means that 10% volume of particles is smaller than this size (27 μm), d50 means that 50% volume of particles is smaller than this size (42 μm) and d90 means that 90% volume of particles is smaller than this size (64 μm).

4. Results and discussion

Olivine (Netherland) and Olivine (Norway) characterisation using Quantitative XRD (X-Ray Diffraction) analysis is discussed. The supplier of these samples has promised to supply olivine; however, these appear as a mixture of olivine and a few other minerals. These samples may be rocks but still, Olivine (Norway) is probably like olivine as it has higher olivine content. This olivine content (62%) almost match lizardite content (61–62%) of the dunite [18] used primarily in my research.
Powders samples were prepared for QXRD analysis of olivine (Netherland) and olivine (Norway). Table 4 shows the analysis. Please refer to earlier publications [2, 10, 13, 14, 18, 19] for detailed description of methods. TGA-MS curves are presented in Figure 14. TGA commonly used in mineral carbonation to obtain yields was coupled with MS (Mass Spectrometer). This enabled us to calculate yields for dunite rock as evolved CO₂ gas and water vapours were measurable. TGA-MS was routinely calibrated. One example of calibration curves is shown in Figure 15.

4.1 TGA-MS analysis of olivine (Norway) and olivine (Netherland)

Olivine (Norway) and Olivine (Netherland) were analysed using TGA coupled with MS (Mass Spectrometer). Results are presented in Figure 14.
These results at least partially confirm that Olivine (Netherland) has more lizardite and is 53.01% as shown in Table 4. This verifies QXRD analysis. Dunite for this research was handpicked from Doonba deposit in vicinity of a small village/town Barbara, close to Tamworth city. Tables 5 and 6 and other following tables present...
phases present in this dunite. Sub 75 μm dunite have 61% lizardite, 29% olivine, 8.3% brucite and 1.3% magnetite [18]. Percentage phases for other dunite fractions (20–45-micron, 45–75 micron and sub 20 micron) are presented in Table 6. An example of calibration curves for TGA-MS calibration is provided in Figure 15, routinely calibration was performed. Dunite is used here for calibration.

### 4.2 Yield calculation from dunite carbonated product

For magnesite yield calculation, please refer to earlier publication [10].

Carbonated products were heated in TGA from 25 to 1000°C and mass losses due to decomposition of different phases present are identified. To identify the evolving gases generated during heating, the TGA-DSC (Setsys Evolution 1200) was coupled with a mass spectrometer (Thermostar Quadrupole). Lizardite decomposes over the same temperature range (300–600°C) as the magnesite and this can introduce a slight systematic error in magnesite yield estimation (leading to an over-estimation of the magnesite yield) unless the mass loss in this period can be quantitatively attributed to loss of H2O or CO2 from the sample. To distinguish between these species, the ion current from the m/z = 44 ion (CO2+) from mass spectrometer was calibrated using sodium bicarbonate samples and a calibration curve for CO2 concentration is obtained, which is used to quantify CO2 mass loss (distinguishing CO2 production from the loss of water vapour which occurs simultaneously) and thus render more accurate estimation of the magnesite yield. CO2 peak areas were calculated using the

| Sr. No | Lizardite | Olivine | Brucite | Magnetite |
|--------|-----------|---------|---------|-----------|
| 1      | –75 μm dunite QXRD | 61      | 32      | 6.4       | 0.37 (difference) |
|        | –75 μm dunite TGA-MS | 61      | 30      | 8.3       | 0.37 (XRD) |

Variation of results with the variation of size, e.g., –75 μm, 20–45 μm, 45–75 μm, –20 μm is interesting.

### Table 5.
Semiquantitative XRD results (3 times XRD repeat, 2 reanalyses).

|                | Lizardite | Olivine | Brucite | Magnetite |
|----------------|-----------|---------|---------|-----------|
| 20–45 μm dunite| 70        | 28      | 1.2     | 0.23      |
| 20–45 μm dunite| 69        | 29      | 1.9     | 0.43      |
| 20–45 μm dunite| 71        | 27      | 1.5     | 0.27      |
| 20–45 μm dunite| 71        | 27      | 0.82    | 0.25      |
| 20–45 μm dunite| 72        | 26      | 1.3     | 0.17      |

### Table 6.
TGA-MS analysis results.
mass spectrometer data and CO₂ mass response is estimated based on the reaction
(2NaHCO₃ → Na₂CO₃ + CO₂ + H₂O). The relationship between peak areas and CO₂
mass loss was linear as shown in the CO₂ calibration curve (Figure 16).

For carbonated samples, the CO₂ peak areas were estimated using MS data and
then these peak areas are used to determine CO₂ mass loss applying the CO₂ calibra-
tion curve. This CO₂ mass loss was used in Eq. (6) to calculate magnesite yield. Eq. (6)
is based on the Gadikota formula (4).

Calculating fraction of magnesium (yₘg) in dunite

\[
\text{Yield (Rx)} = \left( \frac{W_{\text{CO}_2}}{W_{\text{mineral}}} \right) \times 100\%
\]

\[
= \left( \frac{1}{R_{\text{CO}_2}} \right) \times 100\%
\]

\[
= R_{\text{CO}_2} \times \left( \frac{TGA}{100 - TGA} \right) \times 100\%
\]

\[
= R_{\text{CO}_2} \times \left( \frac{TGA}{100 - TGA} \right) \times 100\% \quad (3)
\]

CO₂ storage capacity of dunite = \( \frac{1}{R_{\text{CO}_2}} \) = \( \left( \frac{y_{\text{Mg}}}{MW_{\text{Mg}}} + \frac{y_{\text{Ca}}}{MW_{\text{Ca}}} \right) \times MW_{\text{CO}_2} \) \quad (4)

% of Mg in MgO = \( \left( \frac{MW_{\text{Mg}}}{MW_{\text{MgO}}} \right) \times 100\% = \left( \frac{24.3}{40.3} \right) \times 100\% = 60.3\% \)

% of MgO in dunite = 42.6%

% of Mg in dunite = 60.3\% \times 0.426 = 25.7%

\( y_{\text{Mg}} = 0.257 \) (using this value in equation 5)

Calculating fraction of calcium (yₖa) in dunite

% of Ca in CaO = \( \left( \frac{MW_{\text{Ca}}}{MW_{\text{CaO}}} \right) \times 100\% = \left( \frac{40}{56} \right) \times 100\% = 71.4\% \)
% of CaO in dunite = 0.35%
% of Ca in dunite = 71.4% × 0.0035 = 0.25%

\[ y_{\text{Ca}} = 0.0025 \text{ (using this value in Eq. 5)} \]

\[
\frac{1}{\text{RCO}_2} = \left( \frac{y_{\text{Mg}}}{\text{MW}_{\text{Mg}}} + \frac{y_{\text{Ca}}}{\text{MW}_{\text{Ca}}} \right) \times \text{MW}_{\text{CO}_2} \text{ (equation 5)}
\]

\[
\frac{1}{\text{RCO}_2} = \left( \frac{0.257}{24.3} + \frac{0.0025}{40} \right) \times 44 = 0.468
\]

\[ \text{RCO}_2 = \left( \frac{1}{0.468} \right) = 2.136 \text{ (using this value in equation 4)} \]

\[
\text{Yield (Rx)} = 2.136 \times \left( \frac{\text{TGA}}{100 - \text{TGA}} \right) \times 100\% \quad (5)
\]

\( W_{\text{CO}_2} \) = Weight of CO\(_2\) present in dunite before carbonation.
\( W_{\text{mineral}} \) = Weight of dunite present before carbonation.
\( 1/\text{RCO}_2 \) = CO\(_2\) storage capacity of dunite.
\( y_{\text{Mg}} \) = Weight fraction of magnesium present in dunite which can react with CO\(_2\).
\( \text{MW}_{\text{Mg}} \) = Molecular weight of magnesium (24.3 g/g mol).
\( \text{MW}_{\text{MgO}} \) = Molecular weight MgO (40.3 g/g mol).
\( y_{\text{Ca}} \) = Weight fraction of calcium present in dunite which can react with CO\(_2\).
\( \text{MW}_{\text{Ca}} \) = Molecular weight of calcium (40 g/g mol).
\( \text{MW}_{\text{CaO}} \) = Molecular weight of CaO (56 g/g mol).
\( \text{MW}_{\text{CO}_2} \) = Molecular weight of CO\(_2\) (44 g/g mol).
\( \text{RCO}_2 \) = Mass of dunite required to store unit mass of CO\(_2\).
TGA = CO\(_2\) mass loss from calibration curve.
R\(_X\) = Yield or extent of carbonation.

For a detailed description of materials, analytical instruments and experimental methods, please refer to Chapter 3 of the Ph.D. thesis [2]. Materials, Dunite, heat-activated dunite, heat-transformed dunite, twin sisters mountain dunite, olivine, lizardite and heat-activated lizardite are discussed. Analytical instruments, TGA-MS, XRD, Semi-Quantitative XRD (QXRD), ICP-OES, SEM, EDS, TEM, FTIR and Malvern Mastersizer are discussed. Experimental methods, acid dissolution, regrinding, single-stage carbonation, acrylic reactor testing without temperature and pressure, concurrent grinding both in situ and in operando and two-stage carbonation are discussed. Please refer to these publications for further details [2, 10, 13, 14, 18, 19].

A comparison of elemental composition of dunite by ICP-OES and XRF is provided in Table 7.

4.3 Magnesite yield results using different feedstocks

Magnesite yield results using various feedstocks are presented in Table 8. Few of these results are presented graphically in Figure 17. For already published results, please refer to [2, 19] and [10, 18] and [13, 14]. Soaked dunite especially heat-activated provided the highest yields. This is not evident from literature, especially for heat-activated dunite. However, for raw dunite, some results are presented in Ph.D.
| Element | ICP-OES (wt %) | XRF (wt %) |
|---------|----------------|------------|
| Mg      | 25.8 ± 1       | 25.7       |
| Si      | 12.4 ± 0.3     | 16.6       |
| Fe      | 6.38 ± 0.3     | 6.46       |
| Ca      | 0.373 ± 0.07   | 0.251      |
| K       | 0.04 ± 0.01    | 0.0015     |
| Al      | 0.285 ± 0.04   | 0.171      |

This is a preliminary analysis comparison.

Table 7.
Comparison of elemental composition of dunite by ICP-OES and XRF.

| Sr. No | Particle Size (μm) | Material | Reaction condition | Experiment condition | Reaction time (h) | Yield (%) |
|--------|---------------------|----------|---------------------|----------------------|-------------------|-----------|
| 1      | Sub 75              | dunite   | 185°C, 130 bar, 15% solids, 0.64 M NaHCO₃ | raw                  | 1                  | 24        |
| 2      | Sub 75              | dunite   | 185°C, 130 bar, 15% solids, 0.64 M NaHCO₃ | Raw and regrinding   | 1.5                | 24        |
| 3      | Sub 75              | dunite   | 185°C, 130 bar, 15% solids, 0.64 M NaHCO₃ | Heat-activated (630°C, 4 hrs) | 1                  | 37        |
| 4      | Sub 75              | dunite   | 185°C, 130 bar, 15% solids, 0.64 M NaHCO₃ | Soaked 15 days       |                   | 1         |
| 5      | Sub 75              | dunite   | 185°C, 130 bar, 15% solids, 0.64 M NaHCO₃ | Raw                  |                   | 20        |
| 6      | Sub 75              | dunite   | 185°C, 130 bar, 15% solids, 0.64 M NaHCO₃ | Heat-activated (630°C, 4 hrs) | 1                  | 19        |
| 7      | Sub 75              | lizardite| 185°C, 130 bar, 15% solids, 0.64 M NaHCO₃ | raw                  | 1                  | 2.2       |
| 8      | Sub 75              | dunite   | 185°C, 130 bar, 15% solids, 0.64 M NaHCO₃ | Heat-transformed (800°C, 3 hrs) | 1                  | 18        |
| 9      | Sub 75              | dunite   | 185°C, 130 bar, 15% solids, 0.64 M NaHCO₃ | Heat-transformed (800°C, 3 hrs) | 1                  | 18        |
| 10     | Sub 75              | dunite   | 185°C, 130 bar, 15% solids, 0.64 M NaHCO₃ | Heat-activated (630°C, 4 hrs) | 1                  | 37        |
| 11     | Sub 75              | dunite   | 185°C, 130 bar, 15% solids, 0.64 M NaHCO₃ | Heat-activated (630°C, 4 hrs), 1 month soaked | 1                  | 42        |
| 12     | 20–45               | dunite   | 180°C, 130 bar, 15% solids, 0.64 M NaHCO₃ | Reference            | 2.5                | 5.3       |
| 13     | 20–45               | dunite   | 180°C, 130 bar, 15% solids, 0.64 M NaHCO₃ | Concurrent ground    | 2.5                | 31.6      |
| 14     | 20–45               | dunite   | 180°C, 130 bar, 30% solids, 0.64 M NaHCO₃ | Concurrent ground    | 2.5                | 16.4      |
| 15     | 20–45               | dunite   | 180°C, 65 bar, 15% solids, 0.64 M NaHCO₃ | Concurrent ground    | 2.5                | 12        |
| 16     | 20–45               | dunite   | 155°C, 65 bar, 15% solids, 0.64 M NaHCO₃ | Concurrent ground    | 2.5                | 22.5      |
thesis. Dunite yield calculation is very easy and straightforward, please refer to my Ph.D. thesis publication. Six times higher magnesite yields, or say an increase of 600% [10, 14], or almost two times higher yields in two-stage [13] were achieved using concurrent grinding. Olivine does not accept this much, but still shows some increased trend.

| Sr. No | Particle Size (μm) | Material | Reaction condition | Experiment condition | Reaction time (h) | Yield (%) |
|--------|-------------------|----------|--------------------|----------------------|------------------|-----------|
| 17     | 20–45             | dunite   | 180°C, 65 bar, 30% solids, 0.64 M NaHCO₃ | Concurrent ground   | 2.5              | 10.4      |
| 18     | 20–45             | dunite   | 155°C, 130 bar, 30% solids, 0.64 M NaHCO₃ | Concurrent ground   | 2.5              | 10.9      |
| 19     | 20–45             | dunite   | 180°C, 130 bar, 15% solids, 0.64 M NaHCO₃ | Concurrent ground   | 8                | 62        |
| 20     | Sub 20            | dunite   | 180°C, 130 bar, 15% solids, 0.64 M NaHCO₃ | In operando grinding | 2.5              | 23.6      |
| 21     | 20–45             | dunite   | 180°C, 130 bar, 15% solids, 0.64 M NaHCO₃ | reference           | 8                | 12.3      |
| 22     | 20–45             | dunite   | 180°C, 130 bar, 15% solids, 0.64 M NaHCO₃ | Effect of sampling  | 2.5              | 28.2      |
| 23     | 20–45             | dunite   | 180°C, 130 bar, 15% solids, 0.64 M NaHCO₃ | Effect of sampling  | 1                | 13        |
| 24     | 20–45             | dunite   | 180°C, 130 bar, 15% solids, 0.64 M NaHCO₃ | Effect of sampling  | 0.5              | 9.3       |
| 25     | 20–45             | dunite   | 180°C, 130 bar, 15% solids, 0.64 M NaHCO₃ | Effect of sampling  | 0.05             | 6.3       |
| 26     | Sub 10            | dunite   | 180°C, 130 bar, 15% solids, 0.64 M NaHCO₃ | In operando grinding | 8                | 54.6      |
| 27     | 20–45             | dunite   | 180°C, 130 bar, 15% solids, 0.64 M NaHCO₃ | Concurrent grinding | 2.5              | 31.6      |
| 28     | 20–45             | dunite   | 180°C, 130 bar, 15% solids, 0.64 M NaHCO₃ | Concurrent grinding | 2.5              | 27.2      |
| 29     | 20–45             | olivine  | 180°C, 130 bar, 15% solids, 0.64 M NaHCO₃ | reference           | 2.5              | 0.94      |
| 30     | 20–45             | olivine  | 180°C, 130 bar, 15% solids, 0.64 M NaHCO₃ | Concurrent grinding | 2.5              | 34.1      |
| 31     | Sub 75            | lizardite| 180°C, 130 bar, 15% solids, 0.64 M NaHCO₃ | Concurrent grinding | 2.5              | 5.3       |
| 32     | 45–75             | dunite   | 180°C, 130 bar, 15% solids, 0.64 M NaHCO₃ | Concurrent grinding | 2.5              | 7.1       |
| 33     | 45–75             | dunite   | 180°C, 130 bar, 15% solids, 0.64 M NaHCO₃ | Concurrent grinding | 2.5              | 38.2      |
| 34     | Sub 75            | olivine  | 180°C, 130 bar, 15% solids, 0.64 M NaHCO₃ | reference           | 2.5              | 2.4       |
| 35     | Sub 75            | olivine  | 180°C, 130 bar, 15% solids, 0.64 M NaHCO₃ | Concurrent ground   | 2.5              | 9.2       |

Table 8. Yield results for different feedstocks under various experiment and reaction conditions.
4.4 Semiquantitative XRD results authenticity

Authenticity of QXRD is shown in Table 5.

4.5 TGA-MS results authenticity

TGA-MS results authenticity is excellent. Please see the consistency of magnesite results, which are constant. However, these results have variations as per variation of size fraction.

Error in Brucite calculation due to slight peak overlap.
Error and second option of calculation is shown in Figure 18.

Sr.No. 1. QXRD Calculation details.

| Csi   | Cliz | Coli | Cbru | Cmag | Total |
|-------|------|------|------|------|-------|
| 20.3  | 48.89| 25.287| 5.117 | 0.298| 99.91 |
| Exclude silicon | 61.4 | 31.77 | 6.42 | 0.374 | 100 |

Figure 18.
The left-hand side image graph shows overlap. The right-hand side shows an alternative option.
More results related to this TGA-MS matching with QXRD.

| Sr. | 20–45 μm dunite TGA-MS | Lizardite | Olivine | Brucite | Magnetite |
|-----|------------------------|----------|---------|---------|-----------|
|     | 52                     | 42 (difference) | 5.9 | 0.2 (XRD) |
|     | 20–45 μm dunite TGA-MS repeat | 51 | 43.6 (difference) | 5.27 | 0.2 (XRD) |

| Sr. | 20–45 μm dunite QXRD | Lizardite | Olivine | Brucite | Magnetite |
|-----|----------------------|----------|---------|---------|-----------|
| 1   | 52                   | 42       | 5.8 | 0.2 |
| 2   | 52.6                | 42       | 5.27 | 0.19 |
| 3   | 53                   | 40       | 6.1 | 0.23 |

A very good match between TGA-MS and QXRD results was obtained when using an olivine peak at 17.3°. Brucite also shows a good match. The same peak points were used for all 3 XRD patterns and they are also similar to the -75 μm dunite analysis which provides more confidence in results. Points are slightly changed for 20–45 μm dunite for olivine peaks as these peaks show a slight variation. Results authenticity is excellent.

4.6 Validation of Malvern Mastersizer results

Relationship between d₈₀ (mean particle size) and Malvern mastersizer RPM for olivine (Figure 19). The minimum RPM required for Malvern mastersizer based on feed mean size is given below.

4.7 Validation of olivine yields through QXRD measurements and matched TGA-MS

4.7.1 20: 45 μm olivine crushed carbonated sample QXRD

20–45 μm olivine crushed carbonated reference sample is mixed with 20% silicon and the sample is then scanned for semiquantitative analysis for 3 hrs. This is a reference experiment in which grinding media is not used. The reaction was done at 180°C and 130 bar. Table 9 shows QXRD results matched with TGA-MS results.

![Figure 19](https://via.placeholder.com/150)

**Figure 19.** Relationship between mean particle size and Malvern Mastersizer minimum RPM.

20
4.8 Olivine yield calculation

\[
\text{Mass of } \text{MgCO}_3 = 5.3\% \text{(from QXRD)} = 0.053 \text{ g.}
\]
\[
= 0.053/84 = 0.000628 \text{ moles}
\]
\[
\text{MgCO}_3 \rightarrow \text{MgO} + \text{CO}_2.
\]
\[
\text{Moles CO}_2 = 0.000628 \text{ moles.}
\]
\[
\text{Mass CO}_2 = 0.000628 \times 44 = 0.02766.
\]
\[
\text{Yield} = (0.02766/1) \times 100 = 2.76\% \geq (1\%) \text{ from TGA-MS.}
\]

4.8.1 20–45 \(\mu\)m olivine crushed concurrent ground sample QXRD

20–45 \(\mu\)m olivine crushed concurrent ground sample is mixed with 20% silicon and the sample is then scanned for semiquantitative analysis for 3 hrs. This is a concurrent grinding experiment in which grinding media is used. The reaction was done at 180°C and 130 bar. Table 10 shows QXRD results matched with TGA-MS results.

4.9 Yield calculation

\[
\text{Mass of } \text{MgCO}_3 = 76\% \text{(from QXRD)} = 0.76 \text{ g.}
\]
\[
= 0.76/84 = 0.009 \text{ moles}
\]
\[
\text{MgCO}_3 \rightarrow \text{MgO} + \text{CO}_2.
\]
\[
\text{Moles CO}_2 = 0.009 \text{ moles.}
\]
\[
\text{Mass CO}_2 = 0.009 \times 44 = 0.396.
\]
\[
\text{Yield} = (0.396/1) \times 100 = 39.6\% \geq (34.1\%) \text{ from TGA-MS, reference [14].}
\]

4.9.1 Validation of EDS for 20–45 \(\mu\)m dunite resin embedded samples

Various particles analysis indicate the authenticity of EDS analysis (Figures 20–22). From our earlier articles, a significant difference in morphology of silica-rich layers, especially core and shell part is visible [14]. However, EDS analysis especially silicon shows no significant difference as depicted in above Figures (20–22). However,

| Olivine | Lizardite | Clinohlore | Enstatite | Talc  | Magnesite | Yield |
|---------|-----------|------------|-----------|-------|-----------|-------|
| 20–45 \(\mu\)m olivine concurrent ground experiment | 19% | 0.15% | 0.53% | 0.1% | 3.6% | 76% | 39.6% |

Table 10.
QXRD analysis and yield from QXRD compared with yield from TGA-MS.
as reported earlier, Mg/Si ratio difference [13, 14] is there to confirm the presence of silica-rich layers. This may be taken as one of the key findings of this chapter.

5. Conclusions and recommendations

Suppliers may give wrong materials, but a variety of analyses will determine this. Semiquantitative XRD (QXRD) results authenticity is excellent. TGA-MS results...
authenticity is excellent. No doubt left on TGA-MS and QXRD results matching. ICP-OES results match with XRF results is excellent. EDS results graphically shown are excellent. Routine calibration of measuring instruments must be performed. This very instrument to instrument. Reputed researchers will know the frequency of calibration. The key to calibration is that calibration results match with standard calibration figures/charts or numbers provided by the supplier of the instrument. I recommend contacting the supplier directly or indirectly if calibration curves results are not matching as per intended results. I recommend using pure standard materials for calibration of TGA, MS, ICP-OES, XRD, SEM, EDS, TEM, Malvern Mastersizer, ATR, TPD and other measuring instruments.

Acknowledgements

Muhammad Imran Rashid acknowledges The University of Newcastle, (UoN), Newcastle, NSW, Australia for awarding a Postgraduate scholarship and enabling to use all research facilities especially from Research Division, EMX unit, University of Newcastle, Newcastle, Callaghan Campus, NSW, Australia. Mineral Carbonation International support is beyond imagination. Ms. Kitty Tang support in Malvern Master sizer analysis, Ms. Jennifer Zobec, Ms. Yun Lin and Mr. Huiming provided inevitable support in XRD, QXRD, SEM/EDS/EDX, Simulated QXRD, and TEM. Mr. Scott Molloy support in equipment installations, modifications and difficult amendments is unforgettable. Mr. Glenn Bryant support in ATR analysis and other analyses is highly appreciated. Ms. Jane Hamson support in initial trials of TGA and ICP-OES results is fundamentally acknowledged. Mr. Scott Molloy also helped with TGA-MS especially MS calibrations. His support keeps my work continuous. My advisor Dr. Geoff Brent imaged me with his industry knowledge and capabilities. Fellow students are also acknowledged. Everyone supported me from the heart. I enjoyed cultural diversity and especially food and particularly Lamb.
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