How Ph.D. Students Start a Research

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ABSTRACT

In education pedagogy, it is necessary to prioritize tasks and jobs. Learn operating the reactor or required apparatus but most important first. Regarding measuring techniques (PSD measurement, pH measurement, XRD, SEM/EDS), learn more basic and fundamental techniques first followed by complimentary techniques. Regarding mineral carbonation, operation of reactor is a basic need followed by particle size measurement using Malvern Mastersizer and powders samples XRD analysis and identifying the phases and verifying those phases through TGA analysis. This article illustrates author personal research of 4 initial 4 months duration. Month wise progress is presented in this article in an interesting way.

Keywords: RDH (Research Degree Higher); Ph.D. research start-up; learning measuring techniques; complimentary techniques; mineral carbonation.

1. INTRODUCTION

Reduction in greenhouse gases deemed necessary as their concentration rise sharply from 280 ppm to 419 ppm (1975-2021). Mineral carbonation [1-14], geological storage, oceanic storage and biomineralization as well as chemicals production especially fuels are basic technologies in use. Mineral carbonation is a
permanent fixation of CO₂. A case report of the 4 months research startup which can be considered as author note is demonstrated here. Data and graphs may have been rough but illustrate the future direction of research progress.

2. MATERIALS, METHODS AND EXPERIMENTAL SET-UP

Materials, Methods and experimental set-up is already discussed and shown in earlier publications [1,9,12,13,15,16].

3. RESULTS AND DISCUSSION

Results and discussions are presented in this section. Month wise activities are presented for Month 1, Month 2, Month 3 and Month 4.

3.1 Month 1 Activities

3.1.1 Weekly report

Summary of weekly activities: The main activities done in the last week are described below:

1. Prepared the 15% and 20% dunite slurry by wet grinding in the ball mill for reactor operation
2. Completed online XRD training for qualitative XRD analysis and obtained booking for induction/training/sample analysis
3. Studied quantitative XRD analysis methods and especially fitting full diffraction patterns method which looks more suitable for quantitative calculations.

The main activities planned for next week are:

1. To run the reactor for 15 wt%, 20 wt% and 25 wt% Dunite slurry
2. Do the in-situ pH measurements and analyse to check whether NaHCO₃ is consumed in the reaction or not
3. Dry the slurry for XRD analysis

Summary of weekly activities for next week:
The main activities done in the last week are described below:

4. Performed the carbonation reaction for 15 wt% dunite slurry at 600 rpm, 130 Bar and 185 °C for 1 h

5. Perform three pH measurements, when experiment started, it was 10, dropped to 8.3 after 30 minutes and to 8.0 after 60 minutes, indicating a minor amount of NaHCO₃ is consumed
6. PSD by Malvern Mastersizer (Tests were conducted twice and average taken)

The main activities planned for next week are:

1. To run TGA to calculate the conversion for the reaction
2. Perform XRD analysis of the feed material and reaction product to check which phases are present, it will also provide information that whether feed material have CaCO₃ or FeCO₃ and will further clarify either lizardite react or not
3. Perform ICP-OES analysis of the supernatant solution after the reaction, it will provide information about concentration of magnesium or calcium and other minerals (including silicon) which have not reacted and yield the solid phase.

3.2 Requirements

1. May we can get some more olivine rich material, because Lizardite (60%, as per optical microscopy) in this Dunite will not react without heat treatment, so yield is expected to be low. Later published research indicated this fact [1, 15].
2. To measure amorphous silica, Reitveld Refinement method will not work, because silica does not yield any peaks in XRD. The suitable method for this is fitting full diffraction pattern using FULLPAT. But we need pure individual minerals (few gram) for this method. Pure olivine, lizardite, magnesite (MgCO₃), siderite, brucite, clinopyroxene, amorphous silica, magnetite (Fe₃O₄) etc are present as phases in XRD analysis. Corundum or silicon demanded.
3. To measure only lizardite, corundum reference material is required for quantitative XRD (using intensities comparison method). Our research group and especially my published research used silicon as a reference material [1, 9, 12, 13, 15, 16].
3.3 Month 2 Activities

3.3.1 Weekly reports (4 reports)

Summary of weekly activities: Grinded, 1.1 kg Dunite several times in roller ball mill and got two fractions of fine material, PSD analysis by Malvern Mastersizer report, 30 µm average size.

Operated TGA for reactor product from 25 °C to 800 °C @ 10 °C/min. Two mass losses were observed, the later looks due to the CaCO₃ decomposition. Initially a gradual mass increase was observed. Which can be attributed to water mass loss or bit later to brucite decomposition material [1, 9, 12, 13, 15, 16].

3.3.2 Summary of weekly activities

1. Regrind the sub 100 µm material in a ball mill for two hours and perform PSD analysis.
2. Operate the reactor at 128 Bar and 185 °C for 60 min and samples after 15 min were taken. Then slurry was removed, solids separated and regirnding was done for 20 min in ball mill (MTI Corporation, USA) using twenty five balls of each three smallest sizes. Then again reaction was done at 128 Bar and 185 °C for 30 min and samples were taken. This is a regirnding after the first phases of reaction, published results can be seen here [15]. Pressure variations were observed.

3.3.3 Summary of weekly activities

I made few important observations which are not seen in the literature till date (October 2014).

Decrease in particle size was observed during the reaction in initial hour because of particles dissolution, but no increase in particle size at end of reaction. When I regrind the material after 60 min, particle size decreased and I again performed the carbonation reaction for 30 min. This time at end of experiment, an increase in particle size was observed. I postulate (can be verified by SEM) that after the first reaction, the magnesite formed has acted as seed particles for the next 30 min reaction, that’s why there is increase in size after the reaction. I expect that relatively yield will be higher in the reaction from 60-90 min because of inherent magnesite seeding (related research is here [17]. I expect that if we run the reaction after regirnding for long time, Yield/conversion will further increase rapidly. Hence, yield can also be increased more with seeding initial first hour reaction. One other important observation is that increase in particle size was more for 20 wt % solids slurry than 15 wt% solids slurry suggesting more yield for 20 wt% solids slurry.

Test 2 conditions, 15 wt% solids, 0.64 M NaHCO₃, 128 Bar and 185 °C for 60 min and samples after 15 min were taken. Then slurry was removed, solids separated by gravity setting and regirnding was done for 20 min in ball mill using twenty five balls of each three smallest sizes. Then again reaction was done at 128 Bar and 185 °C for 30 min and samples were taken after 15 min. Published and improved results can be seen here [1].

Test 3 (20 wt% solids, 0.64 M NaHCO₃, 128 Bar and 185 °C). Operated reactor for 60 min and samples after 15 min were taken. Before taking the sample, Syringe Pump was started and reactor was slightly pressurized. After 60 min, reaction was stopped and slurry removed, solids separated, and regirnding was performed for 20 min in ball mill using twenty five balls of each three smallest sizes. Then again reaction was performed at 128 bar and 185 °C for 30 min and samples were taken after 15 min and before taking the sample reactor was slightly pressurized with Syringe Pump. Samples for particle size distribution were taken by stirring the slurry before sampling. Soon later, Syringe Pump was replaced with CO₂ booster compressor, both results were matching as working pressure was same.

3.4 Month 4 Activities

3.4.1 Weekly reports

Summary of weekly activities: I Run TGA many times with Calcium oxalate hydrate (99 % pure) to calibrate it.

Results are shown in Table 8.

1. Done ICP-OES analysis for test 2 (15 wt% solids, 185 °C, 128 bar) supernatant to measure concentrations of different metals.
2. Done ICP-OES analysis for test 3 supernatant.

Month 4 of Research Start-Up: Activities performed are listed below.
3.4.2 Weekly reports

1. Material was analysed by XRD technique
2. Feed material was analysed to identify the phases present. Test 2 (15 wt % solids slurry carbonated after 60 min) before regrinding and after regrinding was analysed. Test 2 carbonated product top and bottom surface layer was analysed separately.

Table 1. PSD analysis

| Test No | Particle Size Distribution before | Particle Size Distribution after |
|---------|----------------------------------|---------------------------------|
|         | \(D_{90}\) \(\mu m\) | \(D_{50}\) \(\mu m\) | \(D_{10}\) \(\mu m\) | \(D_{90}\) \(\mu m\) | \(D_{50}\) \(\mu m\) | \(D_{10}\) \(\mu m\) |
|         | 34.82 | 7.96 | 1.64 | 30.4 | 10.11 | 1.75 |

Table 2. PSD analysis

| Result Units | Volume diameters (average particle size, \(d_{10}\), \(d_{50}\), \(d_{90}\)) |
|--------------|---------------------------------------------------------------|
| Dx (10)      | 5.08 \(\mu m\)                                              |
| Dx (50)      | 29.1 \(\mu m\)                                              |
| Dx (90)      | 70.0 \(\mu m\)                                              |
| Dx (10)      | 2.25 \(\mu m\)                                              |
| Dx (50)      | 20.5 \(\mu m\)                                              |
| Dx (90)      | 73.4 \(\mu m\)                                              |

Table 3. PSD analysis

Dunite slurry (15 wt % solids) at 600 rpm

| Time (min) | Temperature \(^{\circ}C\) | Pressure Bar | pH |
|------------|---------------------------|--------------|----|
| 0          | 185                       | 128          | 9.0|
| 10         | 185                       | 128          |    |
| 20         | 185                       | 124          | 15 min 7.7 |
| 30         | 185                       | 120          | 8.0 |
| 40         | 185                       | 128          | 45 min 8.0 |
| 50         | 185                       | 124          |    |
| 60         | 185                       | 123          | 8.20|
| Regrinding 20 min |                      |              |    |
| 0          | 185                       | 128          |    |
| 70         | 185                       | 126          |    |
| 80         | 185                       | 121          | 75 min 7.8 |
| 90         | 185                       | 120          | 8.0 |

*This may be raw data, but is shown as observed. Later experiments may have improved the results*

Table 4. PSD analysis

| Particle Size Distribution | \(D_{90}\) \(\mu m\) | \(D_{50}\) \(\mu m\) | \(D_{10}\) \(\mu m\) |
|----------------------------|----------------------|----------------------|----------------------|
| Initial feed               | 54                   | 16                   | 2.4                  |
| After 60 min reaction, Before regrinding | 49                   | 11                   | 3.3                  |
| After regrinding           | 24.5                 | 6.7                  | 2.3                  |
| End of experiment, after 90 min reaction | 31.5                 | 8.8                  | 3                    |

Table 5. Test 2, Test repeated for particle size distribution

| Particle Size Distribution | \(D_{90}\) \(\mu m\) | \(D_{50}\) \(\mu m\) | \(D_{10}\) \(\mu m\) |
|----------------------------|----------------------|----------------------|----------------------|
| Initial feed               | 54                   | 16                   | 2.4                  |
| After 60 min reaction, Before regrinding | 49                   | 11                   | 3.3                  |
| After regrinding           | 28.8                 | 7.65                 | 2.41                 |
| End of experiment after 90 min reaction | 34.2                 | 9.06                 | 2.96                 |
Fig. 1. PSD analysis

Fig. 2. PSD analysis
Table 6. Reactor operating conditions and pH analysis

| Time (min) | Temperature °C | Pressure Bar | pH  |
|------------|----------------|--------------|-----|
| 0          | 185            | 128          | 9   |
| 10         | 186            | 127          |     |
| 20         | 185            | 128          | 15 min 8.00 |
| 30         | 185            | 127          | 8.00 |
| 40         | 185            | 127          | 45 min 7.7 |
| 50         | 185            | 127          |     |
| 60         | 185            | 127          | 8.2  |

Regrinding 20 min
| Time (min) | Temperature °C | Pressure Bar | pH  |
|------------|----------------|--------------|-----|
| 0          | 183            | 126          |     |
| 70         | 185            | 125          |     |
| 80         | 185            | 126          | 75 min 7.6 |
| 90         | 185            | 124          | 8.00 |

*pH* was almost constant during the experiment. Slight variation is due to pressure fluctuation while sampling and pressurizing before sampling. Hence, NaHCO₃ was not consumed in the reaction.

Table 7. Particle size distribution Test 3

| Particle Size Distribution | D₉₀ µm | D₅₀ µm | D₁₀ µm |
|---------------------------|--------|--------|--------|
| Initial feed              | 54     | 16     | 2.4    |
| After 60 min reaction, Before regrinding | 44.1   | 10.2   | 3.22   |
| After regrinding          | 37.4   | 8.8    | 2.8    |
| End of experiment after 90 min reaction | 52.4   | 12.6   | 3.26   |

Table 8. TGA calibration

| Step | Reaction | Theoretical Mass loss % | Measured Loss Test 1 15 °C/min % | Measured Loss Test 2 5 °C/min % | Measured Loss Test 3 10 °C/min % |
|------|----------|-------------------------|-----------------------------------|----------------------------------|----------------------------------|
| 1    | CaC₂O₄ + H₂O → CaC₂O₄ | 12.33                   | 10.785                            | 11.73                            | 10.2                             |
| 2    | CaC₂O₄ → CaCO₃ + CO    | 19.17                   | 18.3                              | 18.7                             | 18.8                             |
| 3    | CaCO₃ → CaO + CO₂      | 30.12                   | 31.4                              | 30.3                             | 31.3                             |

Literature Peak temperature with 10 °C/min
| Temperature | Measured Loss Test 1 % | Measured Loss Test 2 % | Measured Loss Test 3 % |
|-------------|-------------------------|------------------------|------------------------|
| 478         | 490                     | 472                    | 495                    |
| 744         | 720                     | 698                    | 735                    |

Table 9. Matched Phases

| Ref. Code | Compound Name | Chemical Formula | Chemical Name |
|-----------|---------------|------------------|---------------|
| 96-900-0167 | Forsterite | Mg₁.00 Mg₇.00 Si₄.00 O₁₆.00 | Forsterite |
| 96-900-6398 | Olivine | Ca₀.93 Mg₇.00 Si₄.00 O₁₆.00 | Olivine |
| 96-900-1639 | Lizardite | Si₂.00 Mg₃.00 O₉.00 H₄.00 | Lizardite |
| 96-100-0055 | Brucite | Mg₁.00 O₂.00 H₂.00 | Brucite |
| 96-900-2325 | Magnetite | Fe₂₄.00 O₃₂.00 | Magnetite |

Table 10. Matched phases

| Ref. Code | Compound Name | Chemical Formula | Chemical Name | Mineral Name |
|-----------|---------------|------------------|---------------|--------------|
| 96-900-5920 | Olivine | Mg₁.00 Mg₇.00 Si₄.00 O₁₆.00 | Olivine | Olivine |
| 96-900-0974 | Magnesite | Mg₆.00 C₆.00 O₁₈.00 | Magnesite | Magnesite |
| 96-900-2324 | Magnetite | Fe₂₄.00 O₃₂.00 | Magnetite | Magnetite |
| 96-900-4510 | Lizardite-1T | Mg₃.00 Si₂.00 O₉.00 | Lizardite-1T | Lizardite-1T |
| 96-900-5435 | Clinopyroxene | Na₂.00 Ca₂.00 Fe₄.00 Si₈.00 O₂₄.00 | Clinopyroxene | Clinopyroxene |
Fig. 3. Feed material

Fig. 4. Test 2 before regrinding
Fig. 5. Test 2 after regrinding

Table 11. Matched phases

| Ref. Code   | Compound Name | Chemical Formula      | Chemical Name  | Mineral Name |
|-------------|----------------|-----------------------|----------------|--------------|
| 96-900-5857 | Olivine        | Fe0.76 Mg7.24 Si4.00 O16.00 | Olivine        | Olivine      |
| 96-900-0974 | Magnesite      | Mg6.00 C6.00 O18.00   | Magnesite      | Magnesite    |
| 96-900-1779 | Lizardite-1T   | Si2.00 Mg3.00 H6.00 O9.00 | Lizardite-1T   | Lizardite-1T |
| 96-900-2324 | Magnetite      | Fe24.00 O32.00        | Magnetite      | Magnetite    |

Table 12. Matched phases

| Ref. Code   | Compound Name | Chemical Formula      | Chemical Name  | Mineral Name |
|-------------|----------------|-----------------------|----------------|--------------|
| 96-900-5857 | Olivine        | Fe0.76 Mg7.24 Si4.00 O16.00 | Olivine        | Olivine      |
| 96-900-0973 | Magnesite      | Mg6.00 C6.00 O18.00   | Magnesite      | Magnesite    |
| 96-900-1779 | Lizardite-1T   | Si2.00 Mg3.00 H6.00 O9.00 | Lizardite-1T   | Lizardite-1T |
| 96-900-2324 | Magnetite      | Fe24.00 O32.00        | Magnetite      | Magnetite    |
| 96-900-6777 | Periclase      | Mg4.00 O4.00          | Periclase      | Periclase    |
### Table 13. Matched phases

| Ref. Code | Compound Name  | Chemical Formula          | Chemical Name  | Mineral Name |
|-----------|----------------|---------------------------|----------------|--------------|
| 96-900-5857 | Olivine       | Fe0.76 Mg7.24 Si4.00 O16.00 | Olivine        | Olivine      |
| 96-900-0974 | Magnesite     | Mg6.00 C6.00 O18.00        | Magnesite      | Magnesite    |
| 96-900-4509 | Lizardite-1T  | Mg3.00 Si2.00 H4.00 O9.00 | Lizardite-1T   | Lizardite-1T |
| 96-900-2324 | Magnetite     | Fe24.00 O32.00            | Magnetite      | Magnetite    |
| 96-900-6475 | Periclase     | Mg4.00 O4.00              | Periclase      | Periclase    |
| 96-900-3800 | Magnesioferrite | Mg8.00 Fe16.00 O32.00    | Magnesioferrite | Magnesioferrite |
Fig. 7. Test 2 bottom layer
Fig. 8. Summed pattern for XRD results
4. CONCLUSION AND RECOMMENDATIONS

Increase in particle size is high in 60-90 min reaction and needles were observed after 90 min [1, 15], yield is low after first 60 min of reaction, hence olivine serpentinization may be occurring in the 60-90 minute reaction. It means lizardite seeding effect after regrinding. But why lizardite less at end of reaction, may be in second part of reaction, olivine is only serpentinizing to lizardite, not to magnesite. While grinding converted the lizardite to amorphous form or another form, hence it may started reaction or may be its amount is high and this reaction lizardite to magnesite has become more feasible. But yet its reactivity is less, so it reacted less, but still sufficient enough that its amount reduced from the amount present in the feed. Quantitative XRD or uniform grinded particles can tell the truth.

Nucleation and crystal growth seen on olivine surface after 1 h, fibres and needles start appearing on the olivine particles. Lizardite supersaturation level was achieved, may be nucleation started on olivine particles, which probably were broken to some extent by regrinding, but again major nucleation of serpentine on olivine surface. The morphology and sorting of the serpentine phases through the reaction cell suggest that crystallization of lizardite was controlled by heterogeneous nucleation on olivine under supersaturated conditions, and crystallization of chrysotile by nucleation on lizardite at higher levels of supersaturation [18], may be silica-rich layer they say is basically a lizardite formed from serpentinization of olivine, which when super-saturated cover the olivine surface and avoid its further reaction [1,9,12,13]. Hence, may be in next time, this lizardite is reacting instead of olivine. if lizardite is in a high quantity to become super-saturate quickly. After first hour of reaction, Lizardite settle on top and olivine settles on bottom, so may be separate lizardite from the top, after drying this slurry or without drying and heat activate only lizardite. No need to heat activate olivine, this
will save energy. Regrind olivine or both olivine and lizardite, mix them and again run the reaction.

One other important analysis is, may be magnesite is settling at the bottom, so doing seeding, increase magnesite cubes in size, now separate magnesite from bottom. This can save energy for regrinding, because we will not be regrinding the magnesite our product. May be use this magnesite, not 100% pure to seed next reaction. May be two reactor are running or two reactor can be used, the product from first is separated and then second reactor is running, with seeding. May be first reactor with seeding in batch mode, which is seeded by its own produced magnesite. Sell spare magnesite. Test these core concepts in pilot plant [10].

From particle size measurement, major increase in particle size because of this serpentinized lizardite formation was found. Further investigations by SEM and XRD were performed. Lizardite can be separated, needles were observed at the top and not at the bottom layer. May be we can do more research to separate lizardite (non-reactive) from olivine to made usage of dunite as an alternative feedstock. Similar happen in XRD, high peak intensity from top layer rather than bottom layer. Needles not observed after initial first hour reaction. More research required to figure out conditions under which this serpentinization can be avoided which seem to cause a major hinderance in utilization of worldwide dunite reserves (Chapter 8, silanol nests formation mechanism, research required [1]). Brucite was completely dissolved and converted to magnesite. It was not detected in XRD analysis of products and in TGA analysis no mass loss was determined.

COMPETING INTERESTS

Author has declared that no competing interests exist.

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