

Analysis of Cementing Carbonation During CO₂ Sequestration

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Abstract. Effects of global warming are well-known to be caused by emissions of greenhouse gases. As an effort to alleviate the gases production, Capture and Sequestration (CCS) is implied in oil and gas practices where carbon dioxide gas (CO₂) is captured from different emission sites and injected into geological formations. However, problems arised in sequestration projects as exposure of CO₂ degraded the Portland-based cement in the wellbore. Consecutively, the cement cracked and leakage of CO₂ contaminated the underground drinking water. Hence, this paper aims to understand the physical and chemical reaction between CO₂ and Class G cement further. Class G cement was obtained from Schlumberger Kemaman Supply Base and tested with water according to American Petroleum Institute (API) standard which water to cement ratio was set to 0:44:1. All of the samples were exposed to CO₂ for six, eight and ten days accordingly and analyzed with unconfined compression, Field Emission-Scanning Electron Microscope (FESEM) and X-Ray Diffraction (XRD) testings. Ten days of CO₂ exposure towards Class G cement showed the lowest compressive strength as to be compared to other samples. Carbonation reaction between water and CO₂ was diagnosed to take place causing the cement to loose its strength. FESEM analysis on the other hand showed that the surface of the sample were uneven with long-shaped crystals with supporting XRD data presented large number of calcium carbonate ((Ca(CO)₃). Nevertheless, the unexposed sample showed the opposite result with highest value of compressive strain and large number of calcium hydroxide ((Ca(OH)₂).

Keywords: Cement Carbonation, Mechanical Strength, Carbon Dioxide Sequestration

1. Introduction

From the beginning of the New Era Revolution in 18th century, the world has seen a sharp increment of energy consumption by means of hydrocarbon burnings which consequently causes dramatic increment of CO₂ being released to the atmosphere. Over the past three centuries, statistic showed that the CO₂ content had increased from 280 to 400 parts per million (ppm)¹. The raise of number gave a weighty impact towards mother earth as the atmospheric CO₂ acts as a warming blanket for the earth and traps the solar heat instead of reflecting it back into the space².



To ameliorate this prominent increment on global temperatures and climate changes, human should start reducing the amount of CO₂ being produced. One of the technologies which believed to be able to reduce the amount of CO₂ effectively is CCS³. CCS operates by capturing CO₂ contained underground and injected it into the cement. So far, favourite spot for CO₂ injection is at the depleted oil and gas occurrence. Over geologic times, this gas had been proven to be capable of trapping fluids deep within the formation. With this, a potential avenue for leakage has been created due to the removal of hydrocarbons from these formations via wells.

Despite of the benefits mentioned, wells could still be potentially providing pathway for either hydrocarbon or the gases leakage. Conventionally, material used for well isolation in oil and gas production is Portland-based cement system. As such, G cement is used due to its advantages of being efficient in conventional well construction and also low in cost. However, Class G cement is known to be thermodynamically unstable in CO₂-rich environments⁴. The cement degrades rapidly once being exposed to acid gases by reacting with ((Ca(OH)₂) formed from hydrated calcium silicate phases⁵. Cement degradation will cause a series of serious problems such as loss of well integrity over period of exposure and the contamination of the underground drinking water. Therefore, effects of sequestration used is really a vital concern to be studied on in order to maintain the integrity of the oil-well cement.

In order to do so, analysis of Class G cement carbonation shall be carried out. There are a few studies which had pointed out different degrees of cement degradation depending on the conditions of exposure such as pressure, temperature, CO₂ and water versus/or CO₂ and brine, from very low degradation to the complete loss of cement integrity. It is required that all the wells in carbon sequestration fields should maintain proper sealing abilities to avoid leaking of CO₂ into the underground portable water formations and atmosphere for long time scales⁶.

2. Simulation Work

2.1. Preparation of Class G cement

Cement slurry samples were prepared using Class G Portland cement and a water-to-cement ratio of 0:44:1. Samples were mixed according to API Recommended Practice 10B⁷. Firstly, weight the amount of cement powder and water needed as shown in **Figure 1**. The water then put into the mixer container and set the blender at 4000 RPM. After that, all of the cement powder weighted should be poured slowly into the mixer container to make sure all cement powder mixes evenly in 15 seconds. The blender then was set in 12000 RPM to mix the slurry in 35 seconds. Then the cement slurry is ready to put into the moulds. Secondly, the moulds were prepared in the shape of cube with 150mm x 150mm. All the sides of the cylindrical moulds were greased before filling it with cement slurry. The cement slurries then poured into the moulds and waiting for hardened in 24 hours at standard condition.



Figure 1. Water and cement weighted

2.2. Carbon Dioxide Exposure

After all cement samples were become hardened, all samples except the control sample were exposed to CO₂ gas inside a cylinder. The cement samples were completely sealed after the exposure to prevent contamination or leaking. Next, each of the samples cured for 6 days, 8 days and 10 days for carbonation reaction takes place.

2.3. Compressive Strength

The NL Scientific 3000kN Unconfined Compressive Strength testing machine was employed for the test. The cement samples were tested using a 3000kN capacity compression machine with a 0.60 kN/s constant loads. The result of compressive strength was shown in the monitor next to the machine once any crack or failure occurs. The compressive strength value was indicated at which value the cement fails or can be known as maximum load.

2.4. Morphology Structure and Chemical Composition Analysis

The test is to study the morphology of both cured and uncured samples. All samples were observed under Field-Emission Scanning Electron Microscope (FESEM) and supported by X-Ray Diffraction (XRD) analysis. Sample of cement was crushed into 2mm fragment to verify the distribution of chemical component can be seen by the machine. The samples were coated with gold before putting it under SEM. By using both of this equipment, how the cured cement responded after CO₂ injected can be determined.

3. Results and Discussion

3.1. Compressive Strength at Different Curing Time

The compressive strength of cement samples was measured by according to standard API⁷. This test was done using Unconfined Compressive Strength (UCS) apparatus. The compressive strength needed to test on carbonation cement samples to find out the cement integrity as well as the mechanical strength of cement. Compressive strength of the Class G cement differs in every curing time. In this test, the control sample is expected to have nearly constant compressive strength due to absence of carbonation reaction in the cement sample. The others sample prepared were cured in 6 days, 8 days and 10 days at standard condition. The obtained results from this test are demonstrated in **Figure 2**. After 6 days of curing in carbon dioxide, the first cement sample possessed a little of dropping compressive strength which is still consider as better. At 8 days of curing time, the Class G cement showed lower value of mechanical strength compare to first cement sample. The last cement sample which cured for 10 days will show the

lowest value of compressive strength. This is because prolonged periods of time is the leaching of cementitious material from the cement matrix and causes decrease of compressive strength⁸. **Figure 2** also shows the comparison of data from Barlet-Gouedard, Rimmele, Goffe and Porcherie (2006) and Yang, Yuan, Wang, Zhang and Zhu (2016) with the experimental result. Barlet-Gouedard et al. (2006) proved that Class G cement with 0.44 of water to cement ratio that has cured in reservoir condition (3000psi and 90°C) with wet supercritical CO₂ has not enough resistance. Compare to the original data, the result is more accurately displayed the potential interaction and degradation processes between the rock formation and cement sheath since the experiment is carried out at downhole temperature, pressure and CO₂ conditions⁹. The Class G cement mixed with carbonation resistance additive ACA proved the better result compare to Class G cement cured in standard condition and reservoir condition¹⁰.

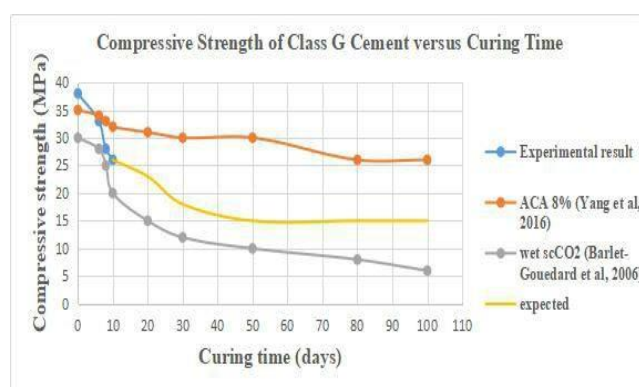


Figure 2. Result of unconfined compressive strength.

3.2. Morphology Structure and Field Emission-Scanning Electron Microscope (FESEM)

Field-Emission Scanning Electron Microscope (FESEM) was run to observe the morphology structure for all cured and uncured cement samples. This machine is used to determine how the cured cement responded to continuous exposure to the CO₂ under standard condition. **Figure 3a** and **Figure 3b** show Class G cement structure under SEM before corrosion respectively. It can be seen that the structure of uncorroded cement sample was dense¹¹. All the compounds showed in the images identify the products form from cement hydration process which can occupy the space or pores between cement grains.

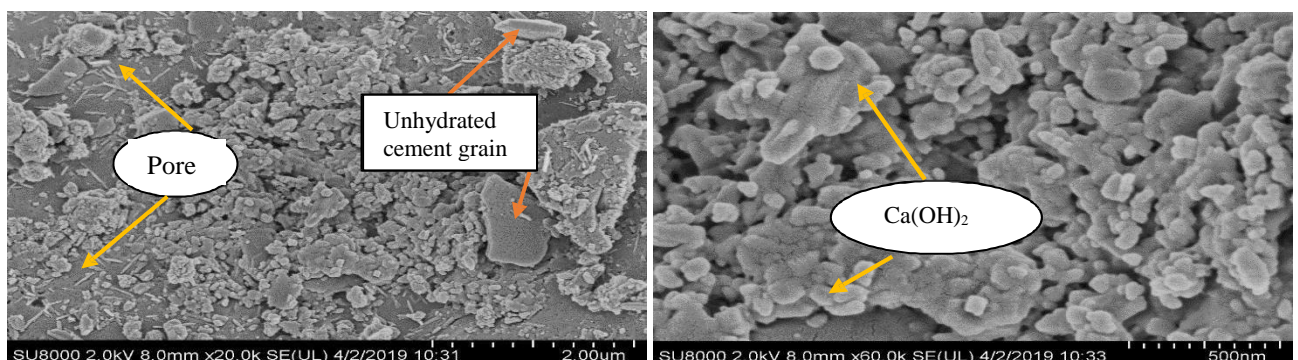


Figure 3a and Figure 3b. FESEM results of uncorroded Class G cement at 20x and 60x magnification respectively.

After the curing period, the rest of the sample also sent together to run FESEM analysis to be compared with the uncured samples. Results shown in Figure 4a and 4b prove that carbonation and

bi-carbonation reaction take places respectively in different period of time. The surface of the cement cured sample was uneven as shown in the figure. Enlarged drawing with 50x magnification revealed that the corrosion products were long- shaped crystals in kinds of configurations. The crystals appeared to be interlinked and interconnected, and there were irregular pits on the surfaces of the crystals. Kinds of microscopic pores were formed in the cement, through which CO₂ could migrate and cause in depth corrosion, and then attack the casings **11**. The results for SEM analysis of cured and uncured Class G cement are supported by the X-Ray Diffractometer (XRD).

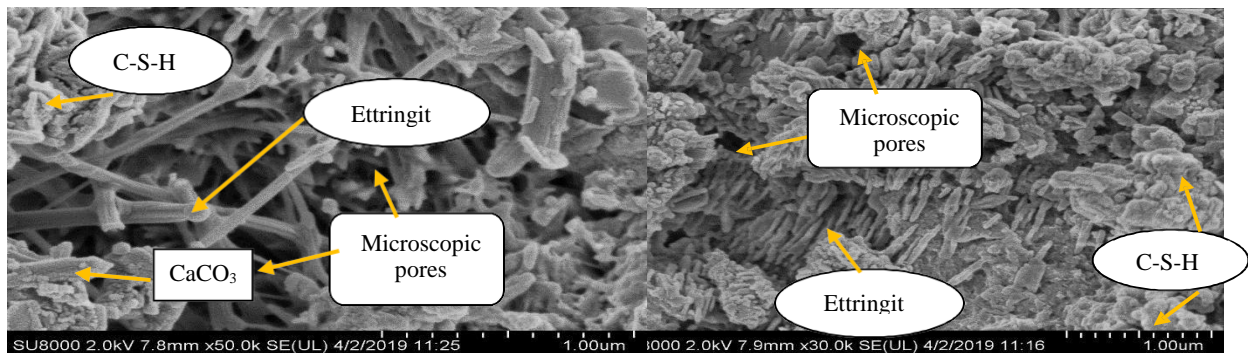


Figure 4a and 4b. FESEM image of cured sample for 10 days for 30x and 50x magnification respectively.

3.3. XRD Analysis

The X-Ray Diffractometer (XRD) analyze the cement samples exposed to gaseous CO₂ under standard condition. This analysis is run to check the chemical composition inside the both cured and uncured cement sample. For the uncured cement sample, the result show that the main composition is Portlandite, calcium hydroxide (Ca(OH)₂) as shown in **Figure**. For the cement samples exposed to gaseous CO₂, as shown in **Figure 6**, the result reveal that the corrosion samples are mainly composed of calcium carbonate (CaCO₃), calcium- silicate-hydrate (C-S-H) and Portlandite (Ca(OH)₂).

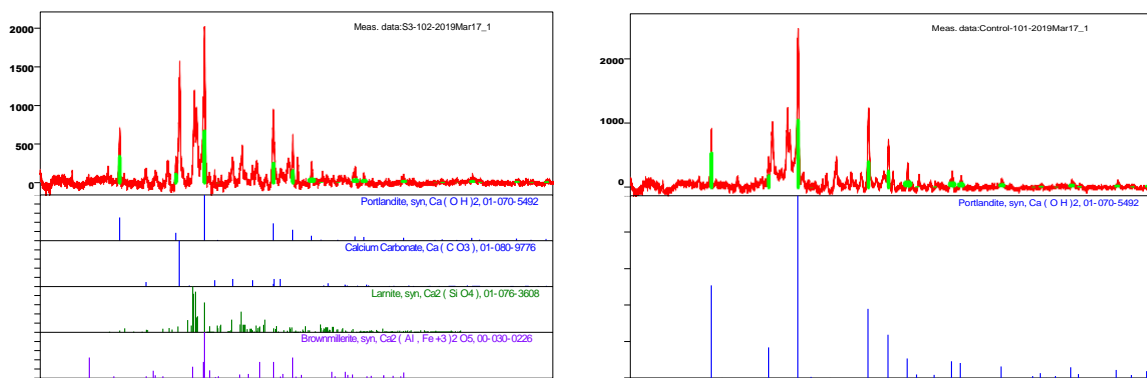


Figure 5. Cured (left) and uncured samples (right).

4. Conclusion

From the result of compressive strength test, Scanning Electron Microscope (SEM) and X-Ray diffraction (XRD) test, bring out some conclusions as per stated below:

1. The uncured Class G cement samples has shown the highest value in Unconfined Compression Strength test which means the degradation has not occur in the cement sample so the sample can withstand the highest force applied on it.
2. The curing period can affect the mechanical strength of cement as well as the cement integrity. For the sample has cured for 10 days, the chemical reaction has reached until bi-carbonation and leads to degradation of cement. The longer the time, the weaker the mechanical strength of the cement due to the release of amorphous silica gel from cement matrix.
3. The image of Field-Emission Scanning Electron Microscope is supported by X-Ray Diffraction (XRD) shows that significance different structure and value in chemical composition of the cured and uncured cement samples.

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