

Cement Degradation Caused Risks for Geological Carbon Sequestration Reservoirs

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Abstract. *Geologic carbon sequestration (GCS), a process to capture and store CO₂ in deep geologic formations, is considered an essential technique to reduce carbon emissions which are a main cause of global warming and climate change. Depleted oil and gas reservoirs are among the most appealing locations for GCS. However, the success of the strategy relies on the long-term integrity of the reservoirs, which is challenged by the carbonation reactions between the main reservoir sealing material, i.e., cement, and the stored CO₂ and then the occurrence of fluid leakage. This work provides a concise review on the degradation of oil well cement and the interface between cement and casing/formation rock, with a special focus on the influence of different factors such as the curing, service conditions and contaminants. The mechanisms and the mitigation methods of the degradation process are also discussed. This work aims to summarise current research progress and identify some remaining challenges for future research.*

Keywords: *Geologic Carbon Sequestration, Supercritical CO₂, Cement Degradation, Integrity.*

1 Background

Geologic carbon sequestration (GCS) is a process in that CO₂ is injected and stored permanently in the subsurface, such as oil/gas reservoirs, non-mineable coal seams, and deep saline aquifers. IPCC and IEA both state that any possible approach to achieving net-zero emissions and tackling climate change cannot exclude the GCS technique (Lin et al., 2022), despite that many methods have been proposed for CO₂ sequestration and utilisation (Meng et al., 2022).

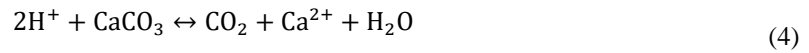
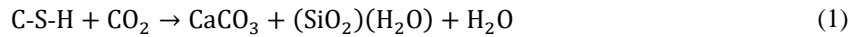
The utilisation of non-producing oil reservoirs/wells for both CO₂ injection and other purposes has long been considered for GCS. The CO₂ captured from the atmosphere and factories are injected into the geological reservoirs deeper than 1 km where the pressure is >7.3 MPa and the temperature is >31.6 °C, resulting in CO₂ gas converting to a supercritical state. Well structures typically include the steel casing and cement ring & plug, securing the sealing condition. However, there are potential pathways for CO₂ to escape from the structure (particularly the interface between cement and casing/formation layer) as its integrity and reliability inevitably deteriorate after a long period in service. Additionally, the downhole temperature, pressure, and brine solution considerably vary with the locations and depths, leading to different degradation risks, e.g., cement carbonation. For instance, the deeper the reservoir, the higher the temperature (e.g., 50 °C at 1 km and 130 °C at 5 km); the brine in sandstone formations has a much lower pH value (pH=2.4) than that in limestone formations (pH=5) where it is calcium-saturated. Therefore, relevant research is necessary to evaluate GCS projects and identify/clarify the carbonation/degradation risks.

Given the progress and prospects of GCS projects, a review of the current research progress is particularly important. This work focuses on summarising the relevant mechanisms and characteristics of well cement carbonation in GCS conditions, the methods adopted for

experiments and numerical simulations, the different risks of the carbonation pathways, the factors influencing the carbonation reactions and some of the mitigation measures. After that, an outlook on this research topic is proposed.

2 Mechanisms of Cement Degradation in GCS Reservoirs

Two major factors affect the cement degradation/carbonation in GCS reservoirs, i.e., the oil well cement (OWC) and the downhole conditions. OWC exists in classes A-J according to several standards, including API 10A, ISO 10426 and GB/T 10238 (SAC, 2015), for different geological conditions and project objectives. Class G and H are the most commonly used. In contrast to ordinary Portland cement (OPC), OWC are better produced for higher compatibility with additives and better storage stability, to maintain the properties such as the ability to withstand high temperatures and pressures, and the resistance to corrosions (Oliveira et al., 2015). But the four basic chemical phases in OPC and OWC remain the same: Alite, Belite, Aluminate and Ferrite. So, the chemical reactions of the cement carbonation in GCS conditions are similar to that in atmospheric conditions to some extent, which involves the two main hydration products: calcium silicate gel (C-S-H) and calcium hydroxide (CH), as shown in Eq.1 and Eq.2. A significant difference is that the carbonation product CaCO_3 further dissolves under the continuous attack from the acidic CO_2 -bearing brines/solutions in GCS conditions (see Eq.3 and Eq.4), which forms a porous structure in hardened cement and results in significant cement degradation.



The main carbonation product calcium carbonate exists in three polymorphs, i.e., calcite, aragonite, and vaterite, among which calcite is more common. But the carbonation products will be slightly different if chemical admixtures are added to cement, given that OWC is rarely used as neat cement. On the other hand, the sealed CO_2 under supercritical states becomes a supercritical fluid with a density of the order of 200~900 kg/m^3 (Gibbins & Chalmers, 2008), with no surface tension and behaving like a gas in terms of compressibility and viscosity. It flows like liquid and is capable to intrude into very fine pores of hardened cement. Besides that, it has a much higher solubility in water in contrast to that in atmospheric condition, allowing a continuous supply of fresh reactants for the carbonation reactions. All these characteristics of supercritical CO_2 result in a much faster diffusion process and carbonation rate (Šavija & Luković, 2016). After the carbonation of OWC, the steel casing is gradually corroded due to the loss of the passive layer. The expansion of the corrosion products will cause cracks and accelerated degradation in return.

3 Carbonation of OWC: Testing, Behaviour and Mitigation

A main function of hardened OWC is to bond geological formations and casing and to prevent potential failure due to geological movement and differences in formation properties. Therefore,

the carbonation resistance of OWC is critical in assessing potential risks. The most ideal test method would be to carry out sampling and analysis of specimens from the field directly. However, many studies are established under laboratory conditions from a more realistic point of view. Specimens were cured according to API standards and carbonation tests were carried out in autoclaves simulating downhole conditions. Various studies tend to cast smaller cement specimens due to the accommodation limit of autoclaves on a lab scale (e.g., cylindrical specimens with a 0.5-inch diameter). Also, different sample preparation methods were adopted, such as drilling from bigger cubes (Rimmelé et al., 2008) or casting in special moulds with covers (Liu et al., 2022), see Fig.1. The smaller specimens also have different curing and carbonation methods. For example, curing 24 h with mould and then curing in an ambient water bath (Lorenzoni et al., 2019) or in a humidity chamber (Elkhadiri et al., 2009), rather than the covering + submerging method in API standard. There is research proving that curing time & procedure, depressurization rate of autoclaves, and even the autoclave design would have a huge influence on the mechanical properties, permeability, etc. (Qin et al., 2021). An example is that the same depressurization rate leads to a lower compressive strength of bigger specimens by 48%~64% and 15% under curing pressure of 50 MPa and 150 MPa, respectively. It may be attributed to longer transport path and higher pore pressure, since the bigger specimen has a 32% higher average elastic modulus and a 56% higher average hardness.

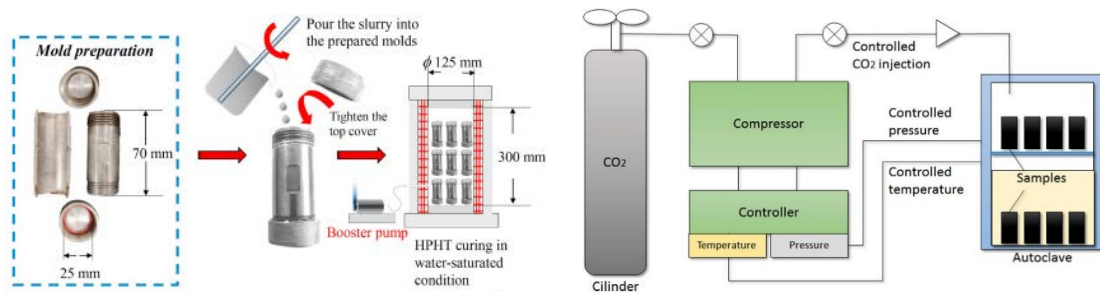


Fig.1 Test procedure in existing research, left: curing with special moulds (Qin et al., 2023), right: autoclave system for carbonation test (de Sena Costa et al., 2018).

In terms of the carbonation behaviour of OWC under supercritical conditions, the carbonated cement showed several distinct layers, which was indicated by studies in both lab-scale and the field. It is due to the high CO_2 concentration and the limited reaction rate caused by the diffusion process. There are currently two most common assumptions about the characteristic layer structures, namely the four-layer and the five-layer assumptions, from the cement core to the exposed surface. Both assumptions consider the innermost part where the mineral composition has not yet reacted as the unaltered core (I). Taking the four-layer assumption (Carroll et al., 2016; Rimmelé et al., 2008) as an example, the typical BSE image is shown in Fig.2. With increasing degradation, CH first dissolves (II), then a dense layer of calcium carbonate forms (III), and finally a leaching decalcification layer with enriched SiO_2 content appears (IV). According to the indentation test results, the hardness of layer III will increase by 50% and that of layer IV will decrease by 60%. The five-layer assumption suggests that layer IV may be further divided into two layers by the colour difference, brown and orange (Duguid & Scherer, 2010). This may be due to the dissolution of brown millerite releasing iron oxide phases and appears to occur only at relatively low pH values (< 2.4) of the aqueous carbonate solution.

Regarding the other influencing factors, the service environment (temperature, pressure, etc.) is no doubt the most important. The higher the temperature and pressure or the lower the pH of brines, the faster the degradation of cement. A 30°C change has a greater influence on the degradation rate than a 1.3 pH change (Duguid & Scherer, 2010); saturated CO₂ brine can increase the degradation rate by 35% compared to supercritical state CO₂ (de Sena Costa et al., 2018). The curing environment is also critical. Temperature (>60°C) has a greater influence on the hydration rates in a short period (10% in 3 days), but the humidity/water is more important for long-term performance (28~90 days), which could lead to a sharp annular carbonation front becoming invisible (Fabbri et al., 2009) or even a significantly reduce of strength by 48%~58% due to non-uniform and weaker formation of C-S-H and differential thermal expansion of cement (Munjaj et al., 2021). In conclusion, the layer structure of OWC carbonated in GCS conditions is well recognized, while it will also be affected by test environments including CO₂ concentration, brines and contaminants, etc.

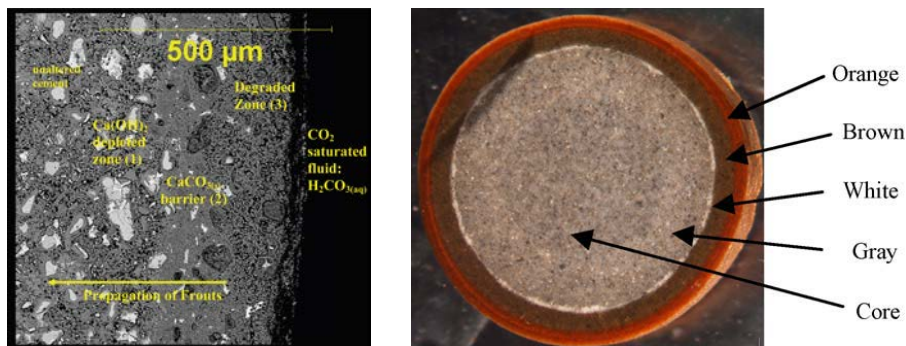


Fig.2 SEM-BSE image for four-layer assumption (left) (Kutchko et al., 2008) and ring pattern for five-layer assumption (right) (Duguid & Scherer, 2010) of neat cement under supercritical carbonation.

Mitigation of carbonation-induced cement degradation has been mainly performed by using additives or industrial wastes. The addition of inert materials (Mn₃O₄, Fe₂O₃, and BaSO₄, etc.) as weighting agents to produce high-density cement slurry seems a good solution to balance the high downhole pressure. Among the different agents, Mn₃O₄ shows the best performance on carbonation resistance (Wu et al., 2023); silica flour can make the cement more tolerant for temperatures over 110°C (Vrålstad et al., 2019). Besides, adding silicon carbide whisker (Li et al., 2019), pozzolanic materials (Abid et al., 2015), carbonic anhydrase (Meng et al., 2022), high Belite cement and quartz sand (Wu et al., 2022) also improve the mechanical properties and carbonation resistance under high temperatures. Nevertheless, it is rather difficult to draw general conclusions on the efficiency of these additives due to the diverse cement compositions used in different studies.

4 Degradation at the Interfaces

The diffusion process of reactants and products may limit the carbonation rate of OWC, preventing it from being the driving cause for well integrity loss. Given the interface of metal-cement and cement-rock are exposed to a complex environment of water, CO₂, gas contaminants (O₂, SO₂, H₂S, NO₂, etc.) and external stresses in many cases, interface is rather easy to be corroded and even fractured, which could be a greater risk. The corrosion products

of the metal-cement interface consist mainly of FeCO_3 , CaCO_3 and some mixed carbonates $\text{Fe}_x\text{Ca}_y\text{CO}_3$ (Dalla Vecchia et al., 2020). Although the corrosion product forms a protective layer that reduces the corrosion rate by a factor of 20, the corrosion rate can still reach tens of mm/year in extreme conditions, which remains an important threat to a GCS reservoir that will last for long time (Carroll et al., 2016), let alone the cracking caused by volume expansion of corrosion. A comprehensive introduction to metal corrosion under supercritical CO_2 (Cui et al., 2019) indicates a “general” type of corrosion appears at the interface, and the corrosion rate first increases and then decreases as the temperature and pressure go up. Additionally, contaminants accelerate the degradation of metal-cement interface, among which NO_2 is the most threatening single contaminant and SO_2 can significantly invalidate the corrosion resistance of Cr. Despite there is research claiming that well-conducted cementing and no interface defect are good enough to prevent the wellbore structure from corrosion (Dalla Vecchia et al., 2020), the mitigation methods at present remain using corrosion inhibitors and cathodic protection, etc. On the other hand, the degradation of the cement-rock interface appears to be more significant than that of the metal-cement interface in the same exposure condition (Nakano et al., 2016). Strong fracturing of both cement and the interface is observed at a macroscopic scale (Jobard et al., 2018), and carbonate crystals precipitate in both rock pores and interface, as shown in Fig.3. The carbonated zone is limited within several millimeters, where Ca^{2+} accumulates by 13%~20% and Mg^{2+} , Si^{4+} and S^{2-} decrease significantly. The crystalline phase, cracking propagation, and carbonation behaviour are all influenced by exposure conditions, such as the Ca/Mg ratio, pH value and contaminants (Nakano et al., 2014, Jobard et al., 2018). Besides, different formation rocks affect the degradation of cement-rock interface as well. For instance, the interface of cement-sandstone has a much higher degradation rate (up to 0.577 mm in three months) while that of limestone-shale is much less significant (Duguid et al., 2011). As for the influence of the external stress, it has already been proved that the confinement of casing has a significant effect on radial and annular damage propagation (Paegle et al., 2019). But more investigations would help to improve the understanding on this matter.

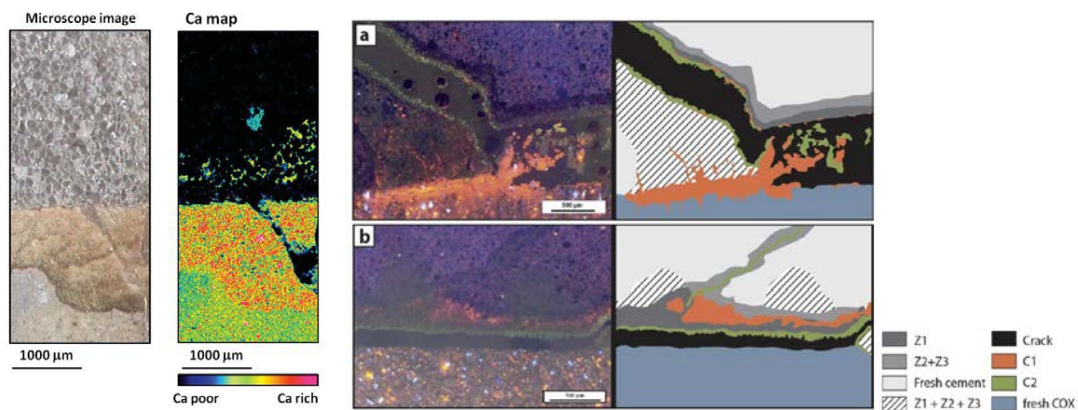


Fig.3 Degradation of cement-rock interface (left): accumulating and leaching of Ca^{2+} (Nakano et al., 2016) (right): the effect of brines on carbonation phases (Jobard et al., 2018).

5 Numerical Simulation Methods

In contrast to experimental research, numerical simulation has special advantages in durability prediction and early detection. Table 1 summarises some of the research work, regarding simulation methods, objectives and conclusions. Most of them are established based on atmospheric carbonation models, revising the characteristic equations of diffusion and reactions to fit the supercritical CO₂ condition. Regarding the simulations of cement degradation, the diffusion process is considered the governing one, which typically can be described by Fick's law. But overall, current studies of carbonation in the GCS context are only based on general formulas and laws, despite of the different focuses and topics. There is still a lack of numerical simulation methods specific for cement carbonation in GCS conditions, whether for durability issues or detection of hidden hazards in wellbore structures.

Table 1. Summary of numerical modelling from studies on supercritical carbonation in GCS.

Ref	Model information	Objectives and conclusions
Rutqvist & Tsang, 2002	Coupled codes of TOUGH2 and FLAC3D for multiphase flow, heat transport and deformations	<ul style="list-style-type: none"> Effect of injected fluid CO₂ on the formation security Indicating the injection zone is the most dangerous part
Kutchko et al., 2008	2 nd Fick law and Elovich equation for degradation under supercritical CO ₂ and CO ₂ -saturated brine	<ul style="list-style-type: none"> Cement degradation Suggesting a great degradation for neat cement is unlikely
Scherer & Huet, 2009	2 nd Fick law and linear CO ₂ solubility in shale with a magnitude of 10 ⁻¹¹ m ² /s	<ul style="list-style-type: none"> Cement degradation Indicating CO₂-saturated brine in the formation may cause several centimeters' carbonation depth in cement
Zhang et al., 2013	Utilising CrunchFlow® and considering the effect of H ₂ S	<ul style="list-style-type: none"> Cement degradation Showing the reaction of pozzolan-amended cement and H₂S leads to the formation of pyrite and ettringite
Xiang et al., 2013	Proposing a six-layer model based on the existing TCPC model	<ul style="list-style-type: none"> Casing degradation Predicting the corrosion rate of casing steel and determining the concentration distribution of each component in the water and product films
Zha et al., 2015	Modifying the multi-factor coupling model by real gas law and Darcy's law driven by pressure difference	<ul style="list-style-type: none"> Casing degradation Revealing the influence of saturation, temperature, and pressure on the carbonation depth

6 Conclusions

- Experimental carbonation studies of OWC often use small specimens (e.g. due to the limited volume capacity of lab autoclaves) with different curing and testing methods compared with that specified in, e.g. the API standard. Because of the difference in the

various adopted test procedures, it is rather difficult to draw general conclusions. Therefore, practical recommendations for the test procedures for small specimens would benefit the research community.

- For cement carbonation in GCS conditions, the mechanisms are rather well explored and the characteristic layer structure is found from samples in both laboratory and the field. The influence of temperature, pressure, brines and contaminants has also been studied. Nevertheless, most of the existing research is based on results obtained under laboratory conditions. The available data for sampling and testing from the field are still limited.
- The degradation at the metal-cement and cement-rock interfaces has been investigated. Compared to the metal-cement interface, a greater risk is found at the cement-sandstone interface under the same exposure environment. Besides, both contaminants and environmental conditions, such as the type of the formation rock, have important influences on the degradation behaviour.
- Numerical simulations specific for cement carbonation problems under supercritical CO₂ conditions are missing, while they hold great potential for research compared to experimental studies.

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