Improving porosity and permeability of sandstone reservoirs for CO₂ storage by enhancing mineral dissolution with bio-based biodegradable chelating agents

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Abstract

Geological storage and mineralization of CO_2 in sandstone reservoirs sometimes faces challenges in reservoir properties and injection technologies, including limited interconnected pore spaces (i.e., effective porosity), permeability, and rock reactivity, as well as difficulties in using seawater for CO_2 capture and uncontrolled carbonation. To address these issues, the authors have recently developed a new approach to CO_2 capture, geological storage, and mineralization, primarily for mafic and ultramafic rock reservoirs, with the innovative use of bio-based, biodegradable chelating agents and seawater. The method involves injecting an acidic chelating agent solution to increase the effective porosity and permeability through chelating-agent-driven enhanced mineral dissolution. Subsequently, the use of alkaline chelating agent-containing seawater improves CO_2 capture and geological storage efficiency by inhibiting carbonate formation and crystallization, thereby maintaining injectivity while providing divalent metal ions for mineralization and further expanding storage space. Finally, control of the CO_2 mineralization process is achieved by adjusting the biodegradation of chelating agents.

The present study aimed to investigate the applicability of this method, especially the acidic chelating agent solution injection part, to sandstone reservoirs through batch experiments using rock powder and flow-through experiments using a rock core. All experiments were conducted at 35° C using GLDA-Na₄ (tetrasodium glutamate diacetate) as a representative of bio-based, biodegradable chelating agents. Batch experiments at pH 1-13 showed that GLDA-Na₄ significantly increased the release rate of various elements from minerals over a wide pH range. Rates were generally higher at lower pH. Importantly, however, the rate for calcium was almost independent of pH and therefore high even at alkaline conditions suitable for CO₂ mineralization. Flow-through experiments at pH 1 showed that a rapid and substantial permeability improvement (e.g., doubling in 3 hours) occurred along with pore formation due to enhanced mineral dissolution. The experimental results suggest that the new CO₂ capture, geological storage and mineralization method can be applied to sandstone reservoirs.

Keywords

CO2 storage, porosity, permeability, sandstone, chelating agent





1 Introduction

To combat climate change, the Paris Agreement calls for limiting the average global temperature increase to $1.5-2^{\circ}$ C relative to pre-industrial levels (Colón-González et al. 2018). To achieve this goal, greenhouse gas emissions must be reduced by 43% by 2030 compared to 2019 levels, which will require scaling up removal and storage to approximately 10 Gt CO₂ per year by 2050 and 20 Gt CO₂ by 2100 (Matter and Kelemen 2009). Among the various options for CO₂ storage, geological storage emerges as a promising means to meet the ambitious global climate goal due to the large storage potential and the abundance of storage reservoirs, including sedimentary formations and unconventional reservoirs (mafic and ultramafic reservoirs) (Kelemen et al. 2019; Ajoma et al. 2020).

However, there are concerns regarding the capacity and injectivity (i.e., ease of fluid flow) of reservoirs for CO₂ storage. First, the interconnected pore spaces (i.e., effective porosity) and permeability of rocks are not always sufficiently high (Callow et al. 2018). Permeability, and thus injectivity, can be further reduced by carbonate precipitation during CO₂ injection (Raza et al. 2017) (Silveira et al. 2022; Alfredsson et al. 2013). Therefore, CO₂ mineralization should be minimized during the injection process. Another concern is the reactivity of the rock for CO₂ mineralization. While CO₂-dissolved acidic water can promote rock dissolution by providing more H⁺, this effect is small at the typical temperatures of reservoirs targeted for CO₂ storage, which range from 20 to 50°C (Snæbjörnsdóttir et al. 2020; Black et al. 2015; Brantley 2008). Finally, the use of large quantities of freshwater for CO₂ mineralization is the priority and CO₂-charged water is injected instead of CO₂ alone. Although the use of seawater instead of fresh water has been proposed, its efficiency is even lower (reduced by about 25%) (Marieni and Oelkers 2021)

The effective use of bio-based and biodegradable chelating agents provides a solution to these challenges. Chelating agents can form complexes with metals over a wide range of pH values. For this reason, they are widely used in industrial, domestic and agricultural applications. They can enhance mineral dissolution by promoting leaching of both metals and silicon (Wang et al. 2022; Takahashi et al. 2023; Wang et al. 2024a), have been used to improve permeability in oil field stimulation, and are potentially promising in geothermal field stimulation (Watanabe et al. 2021; Salalá et al. 2023; Salalá et al. 2024). Among the various types of chelating agents, some are biobased and readily biodegradable, such as glutamate diacetate (GLDA), a derivative of glutamic acid, which is inexpensive compared to other chelating agents and attractive for use in natural environments (van Ginkel and Geerts 2016). Bio-based chelating agents are particularly attractive for CO₂ storage because their production involves the absorption of large amounts of CO₂ from the atmosphere through photosynthesis.

Therefore, we have recently developed an approach for enhanced CO₂ capture, geological storage and mineralization using bio-based and biodegradable chelating agents, primarily for mafic and ultramafic rock reservoirs (Wang et al. 2024b). Figure 1 illustrates the operational processes using GLDA as a representative chelating agent. The process consists of the following steps: First, since the effect of GLDA on mineral dissolution is more pronounced under acidic conditions (Wang et al. 2024a), an acidic GLDA solution is injected to enhance rock dissolution. This accelerates the leaching of divalent metals from minerals for CO₂ mineralization and increases the pore volume, connectivity and permeability of the rock, thereby improving the capacity and injectivity for CO₂ storage. An enhanced CO₂ reservoir can store either CO₂ or CO₂-charged water. To maximize the efficiency of CO₂ capture, storage, and mineralization, an alkaline GLDA seawater solution is proposed for the second step. The alkaline and pH buffering properties of commercial GLDA-Na₄ solution can serve as an effective absorbent for CO_2 capture. The use of seawater as a solvent for GLDA not only overcomes the challenge of significant freshwater consumption, but also provides divalent metals and microbes for CO₂ mineralization and GLDA biodegradation, respectively. In addition, the injection of GLDA solution with CO₂ inhibits mineralization because almost all of the metal ions in the solution are bound to GLDA as chelates and therefore do not contribute to the formation of secondary minerals. The presence of GLDA also inhibits the crystallization of carbonate; even if a small amount of carbonate is formed, it is nano-sized (Moulay et al. 2023). This allows the CO₂ storage process to proceed without significantly reducing the effective porosity and permeability. Injection of the solution may actually increase the effective porosity to some extent through enhanced mineral dissolution. Finally, GLDA biodegradation in the reservoir releases both metal ions (those leached from rocks or in the original seawater) and CO₂ (captured from the atmosphere during GLDA raw materials production), triggering

 CO_2 mineralization. Therefore, the rate of CO_2 mineralization can be controlled by adjusting the rate of GLDA degradation, for example, by changing the number of microbes in the injection fluid or reservoir.



Fig. 1 Diagram of enhanced CO₂ capture, geological storage and mineralization using a bio-based and biodegradable chelating agent, glutamate diacetate (GLDA) solutions (Wang et al., 2024b).

This approach addresses challenges in existing technologies, including potentially low effective porosity, permeability, and rock reactivity, as well as difficulties in using seawater for CO_2 capture and uncontrolled carbonation (referring to the difficulty in controlling the timing of carbonation). The purpose of this study is to investigate the applicability of this method, particularly the acidic chelating agent solution injection part, to sandstone reservoirs through batch experiments using rock powder and flow-through experiments using a rock core.

2 Materials and Methods

2.1 Materials

The sandstone used in the present experiments was obtained from a saline aquifer at a depth of approximately 1.4 km in Niigata, Japan. The major mineral compositions were plagioclase, quartz, zeolite, chlorite, and mica/illite, accounting for approximately 59.1, 24.0, 6.3, 4.8, and 5.0 wt%, respectively. The mineral compositions were determined by X-ray diffraction (XRD; Multiflex, Rigaku, Japan) using Cu $K\alpha$ radiation ($\lambda = 1.54$ Å) operated at 40 kV and 20 mA with a 2 θ step size of 0.02° from 3° to 50°, and an X-ray diffraction analysis software, JADE, from Materials Data, Inc.

The bulk chemical composition of the sandstone was determined by X-ray fluorescence spectrometry (XRF; Rigaku, ZSX Primus IV) and is presented in Table 1. The sandstone was crushed and sieved to obtain the < 100 μ m fractions for the batch dissolution experiment. In addition, a core sample of the sandstone measuring 38 mm in diameter and 34 mm in length was prepared for the flooding experiment. The core sample was measured to have a porosity of approximately 23% at room temperature and atmospheric pressure, and a permeability of 8.40×10^{-16} m² at a confining pressure of 2.76 MPa at 35°C.

For both the batch dissolution and flooding experiments, aqueous solutions of tetrasodium glutamate diacetate (GLDA-Na₄) were prepared at desired combinations of GLDA-Na₄ concentration and pH,

with the pH of these solutions adjusted by the addition of a solution of nitric acid (HNO_3). For the flooding experiment, an aqueous solution of NH_4Cl was also prepared to measure the permeability of the core sample before and after injection of the chelating agent solution.

Component	Content (wt%)
Na ₂ O	2.128
MgO	1.607
Al ₂ O ₃	10.601
SiO ₂	68.278
P2O5	0.094
K ₂ O	3.000
CaO	1.461
TiO ₂	0.529
MnO	0.033
Fe ₂ O ₃	3.409
Total	91.140

Table 1 Bulk chemical composition of the sandstone.

2.2 Powder experiments

A series of batch dissolution experiments were conducted with the sandstone powder. In each experiment, 5 g of sandstone powder was mixed with 100 mL of GLDA-Na₄ solution at the desired combination of pH (1-13) and GLDA-Na₄ concentration (0-20 wt%) in a plastic sample bottle, which was then placed in a thermostatic shaker for continuous shaking. All experiments were conducted at 35° C for 6 hours at a shaking speed of 150 rpm. At the end of the reaction, both the solution and the solid components in the bottle were collected separately using a 0.45 μ m membrane filter.

The collected solution samples were analyzed for concentrations of Mg, Al, Si, K, Ca, Fe, Mn, and Ti using inductively coupled plasma optical emission spectrometry (ICP-OES; Agilent 5100). The mineral composition of the sandstone powder after the experiment was measured using X-ray diffraction and X-ray diffraction analysis software.

2.3 Flooding experiments

3-hour chelating agent flooding experiment was performed three times on the same sandstone core sample using 10 wt% GLDA-Na₄ solution at pH 1 with the experimental system shown in Figure 2. A Viton-sleeved sample with end plugs attached to both the inlet and outlet faces was placed horizontally in a pressure vessel maintained at 35°C using a mantle heater. The sample was then subjected to a confining pressure of 2.76 MPa by pumping water into the vessel at a constant pressure. The permeability of the sample prior to injection of the chelating agent solution was determined using Darcy's law by injecting 5 wt% NH₄Cl solution (brine) into the sample at a constant flow rate of 1 mL/min using a syringe pump. The viscosity of the brine is 0.77 mPa·s. During this process, the brine flowed out of the sample at atmospheric pressure. The chelating agent solution was then injected into the sample at the same flow rate for 3 hours. During this process, the injection pressure becomes higher (up to about 0.7 MPa) than during the brine injection because the viscosity of the chelating agent solution (1.2 mPa·s) is higher than that of the brine. Finally, the brine was injected into the sample again to remove the chelating agent solution and to determine the permeability after injection of the chelating agent solution.

Effluent samples were collected during injection of the chelating agent solution. Their pH and elemental concentrations were analyzed by ICP-OES. X-ray computed tomography (CT) was performed on the sample before and after each chelating agent flooding experiment at a tube voltage of 120 kV, tube current of 150 μ A, and voxel size of 12 × 12 × 12 μ m. Molcer Plus 3-D image visualization and processing software (White Rabbit Corp., Tokyo, Japan) was then used to visualize the distributions of large pores with sizes similar to or larger than the voxel size.

(a) Sandstone core sample

(b) Experimental system



Fig. 2 Sandstone core sample (a) and the experimental system for the flooding experiment (b).

3 Results and Discussion

3.1 Chelating-agent-driven enhanced dissolution of the sandstone

Figure 3 shows GLDA-Na₄ concentration and pH dependencies of elemental concentrations in the solution after the 6-hour batch dissolution experiment on the sandstone powder. The concentration dependence was investigated using 0-20 wt% GLDA-Na₄ solution at 35°C and pH 4, and the pH dependence was investigated using 10 wt% GLDA-Na₄ solution at pH 1-13 at 35°C.



Fig. 3 GLDA-Na₄ concentration (a) and pH (b) dependencies of elemental concentrations in the solution after the 6-hour batch dissolution experiment on the sandstone powder.

Concentrations of many elements increased with increasing concentration of GLDA-Na₄, demonstrating the chelating agent-driven enhanced mineral dissolution (Fig. 3a). Even at the lowest concentration of 1 wt%, enhanced mineral dissolution was evident. However, the intensity of the enhancement was not proportional to the concentration of GLDA-Na₄. In particular, the increase in intensity occurred up to 2 wt%, beyond which the increase became smaller. This means that a similar intensity of enhanced mineral dissolution can be expected over a wide range of GLDA-Na₄ concentrations.

Concentrations of most elements increased with decreasing pH, with only Ca concentration being significantly high at pH 13 (Fig. 3b). Importantly, the Ca concentration remained similarly high at all pH conditions. These results support the validity of the proposed method of enhanced CO₂ capture, geological storage and mineralization through the use of a bio-based and biodegradable chelating agent solution (Fig. 1), where an acidic chelating agent solution is injected to improve reservoir porosity and permeability, while a CO₂-charged alkaline chelating agent solution is injected into a reservoir to facilitate CO₂ mineralization.

Fig. 4 shows changes in the major mineral compositions of the sandstone powder after the 6 h batch dissolution experiments at pH 1 and 8. At pH 8, only plagioclase dissolution was dominant, whereas mica/illite, chlorite, and quartz dissolution were dominant at pH 1. The dissolved quartz probably contained metallic elements as impurities, since chelating agents do not react with Si. These changes in mineral composition are consistent with the fact that more elements showed high concentrations in the solution with decreasing pH (Fig. 3b).



Fig. 4 Major mineral compositions in the sandstone powder before and after the experiments at pH 1 and pH 8.

3.2 Poro-perm evolution of the sandstone by the enhanced mineral dissolution

Figure 5 shows evolution of elemental concentrations and pH of the effluent for each flooding experiment in which 10 wt% GLDA-Na₄ solution at pH 1 was injected into the sandstone core sample under 2.76 MPa confining pressure at 35°C for 3 hours. In the figure, the concentration and pH values at 0 min were obtained for an effluent during brine injection (prior to injection of the chelating agent solution). Note that the scale of the left vertical axis for elemental concentration was reduced for the later experiment.



Fig. 5 Evolution of elemental concentrations and pH of the effluent for each flooding experiment on the sandstone core sample.

The elemental concentrations during injection of the chelating agent solution generally decreased with time, indicating that mineral dissolution became slower with time. However, even in the third experiment, the elemental concentrations were generally much higher than those during brine injection, indicating that chelating-agent-driven enhanced mineral dissolution could occur for a long period of time.

Figure 6 shows the distribution of large pores (> \sim 12 µm) and permeability of the sandstone core sample before and after each flooding experiment. In the distribution of large pores, warmer coloured pores correspond to larger sizes. Since the sample initially contained some macropores (e.g., fractures), large pores were already distributed in the initial state. However, most pores were not visualized due to their small size (i.e., < 2 µm by a mercury intrusion porosimeter).

Before the experiment, the permeability was 8.4×10^{-16} m². After the first experiment, the permeability increased to 2.0×10^{-15} m², or 2.4 times the initial value, within only 3 hours. However, the amount of large pores did not increase; in fact, it appears to have decreased slightly due to the confining pressure. This means that a significant improvement in permeability was achieved by increasing the number of small pores (< ~12 µm), such as the formation of larger pore throats. The permeability further increased to 2.4×10^{-15} m², or 2.9 times the initial value, after the second experiment. Along with this increase in permeability, there was a significant increase in larger pores. However, after the 3rd experiment, although the amount of large pores continued to increase, the permeability did not increase but decreased slightly to 2.2×10^{-15} m². It is likely that some of the pores that contributed to

fluid flow were reduced in size by the rearrangement of sand grains by the repeatedly applied confining pressure, resulting in the slight decrease in permeability. These results suggest that the permeability improvement was mainly due to the formation of larger pore throats, which could be achieved even with removal of small amounts of minerals by a short reaction time, while the porosity improvement was mainly due to formation of larger pore bodies, which requires removal of large amounts of minerals by a long reaction time.



Fig. 6 Evolution of porosity and permeability through the three flooding experiments on the sandstone core sample where the chelating agent solution was injected from top to bottom in the figure.

4 Conclusions

The present study aimed to investigate the applicability of the new CO_2 capture, geological storage and mineralization approach with the innovative use of bio-based biodegradable chelating agents and seawater, especially the acidic chelating agent solution injection part, to sandstone reservoirs through the batch experiments using the sandstone powder and the flow-through experiments using the sandstone core. All the experiments were conducted at 35°C and used GLDA-Na₄ as a representative of bio-based biodegradable chelating agents.

The batch experiments at pH 1-13 showed that GLDA-Na₄ significantly increased the release rate of various elements from minerals over a wide pH range. The rates were generally higher at lower pH. Importantly, however, the rate for calcium was almost independent of pH and therefore high even at alkaline conditions suitable for CO_2 mineralization. The flow experiments at pH 1 showed that a rapid and substantial permeability improvement (e.g., doubling in 3 hours) occurred along with pore formation due to the enhanced mineral dissolution. These experimental results suggest that the new CO_2 capture, geological storage and mineralization method, primarily devised for mafic and ultramafic rock reservoirs, can be applied to sandstone reservoirs.

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