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Energy Procedia 63 (2014) 4536 – 4547

Energy Procedia

GHGT-12

CO_2-DISSOLVED: a Novel Concept Coupling Geological Storage of Dissolved CO_2 and Geothermal Heat Recovery – Part 3: Design of the MIRAGES-2 Experimental Device Dedicated to the Study of the Geochemical Water-Rock Interactions Triggered by CO_2 Laden Brine Injection.

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Abstract

The CO_2-DISSOLVED project aims at assessing the feasibility of the coupling between dissolved CO_2 storage in aquifer and geothermal heat recovery. The MIRAGES-2 experimental setup has been designed to study, at the centimeter scale and under relevant conditions of pressure and temperature, the chemical interactions in the near-injection well area between the reservoir rock, the cement phases, and the corrosive CO_2-rich solution. This original experimental setup allows performing flow-through experiments with continuous in-situ data acquisition of pressure, temperature, flow rate, pH, and dissolved CO_2 concentration. The datasets acquired will be further interpreted with the help of geochemical models, in order to better understand the effects of the key physical-chemical processes involved.

Peer-review under responsibility of the Organizing Committee of GHGT-12

Keywords: experimental design; MIRAGES-2; geological storage; geothermal energy; dissolved CO_2; limestone; class G Portland cement; injection well

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Peer-review under responsibility of the Organizing Committee of GHGT-12
doi:10.1016/j.egypro.2014.11.487

1. Introduction

The geological storage of CO_2 is envisaged to mitigate the anthropogenic greenhouse gas (GHG) emissions in the short term [1-4]. Projects for this geological storage generally plan for its injection under supercritical conditions, thus maximizing the stored quantities that can involve several million tons per year. The CO2-DISSOLVED project [5] studies a different option that consists in combining injection of dissolved CO_2 close to the emitting source and recovery of the geothermal heat from the extracted brine. This approach relies on the geothermal doublet technology (commonly used in the Paris Basin, France), where the warm water is extracted at the production well and the cooled brine re-injected in the same aquifer via a second well (injection well).

Basically, recovering the heat from the extracted brine will rely on the well-known technology of heat exchangers that will not be different from that used in classical low-energy geothermal exploitation facilities. However, adding dissolved CO_2 in the injected brine obviously adds new constraints that should be accounted for in the design and the sizing of the whole system. Indeed the injection of CO_2-rich acidified water is expected to induce an enhanced reactivity at the immediate vicinity of the injection well, particularly in presence of carbonated minerals. Similarly, acidified water will be much more aggressive for the well casing than standard cold brine in classical geothermal doublets. This injection option has been much less studied than the standard injection of supercritical CO_2, so we need to improve our knowledge on these aspects. Specific work, focusing on the near-injection well area and relying on both new experimental and modeling approaches will be carried out in the framework of the CO_2-DISSOLVED project.

The so-called MIRAGES (acronym, in French, for “Radial Model for Greenhouse Gas Injection”) experimental device was initially designed by GeoRessources for application to supercritical CO_2 injection ([6-8]). It mimics an injection well at the 1/20th scale. In this study, we present a new experimental device (MIRAGES-2) that was specifically designed for the injection of dissolved CO_2 under realistic storage conditions (60°C – 120 bar).

Corrosion problems linked to the injection of an aggressive fluid in the well are investigated by using different materials of casing (steel, composite materials).

The implementation of the test bench and the data acquired during the experiment will allow to understand the impacts within the reservoir and in the well (corrosion issues), and their consequences in terms of both integrity and lifetime of the coupled CO_2 storage/injection system. The different experiments will help validating the numerical simulations and the final objective will be then to define possible injection scenarios which are optimized for the best coupling between safe CO_2 storage and efficient heat recovery.

This paper presents the MIRAGES-2 setup through a first experiment performed with a Lavoux limestone, a class G Portland cement and a well casing made of stainless steel. This experiment helps validating the experimental setup and the ability to perform the desired measurements.

2. Experimental design and procedure

2.1. Sample description

The Lavoux limestone is a reservoir rock considered as the natural analog of the Bathonian oolitic limestone (Oolithe Blanche formation) of the Paris Basin. The Dogger aquifer of the Paris Basin was identified as a potential host formation for supercritical CO_2 storage ([9, 10]) and has been used for geothermal projects for more than 30 years. This first sample was then selected as being representative of a potential good candidate area for a future implementation of the CO2-DISSOLVED concept.

The porosity of the Oolithe Blanche formation ranges from 16 to 23% and the permeability varies between 0.1 and 10 mD. The Lavoux limestone is composed of 98% of calcium carbonate (calcite and low magnesium calcite, [11]).

The core assemblage used to reproduce a scaled-down injection well (Fig. 1) consists of either a metallic or a non-metallic tube (length = 119 mm, internal diameter = 4.5 mm, external diameter = 6.35 mm) screwed to a Teflon ring (diameter = 25 mm, thickness = 2 mm) and cemented to the core plug (external diameter = 100 mm, length = 100 mm) with a class G Portland cement. A piece of composite material that could be used instead of steal for the well casing due to a better resistance to corrosion issues, can be placed at the injection zone or at the interface between the reservoir and the cement.

Cement slurry is prepared by adding 92 g of powder cement to 349 g of demineralized water according to the API-ISO 10426 A norm.

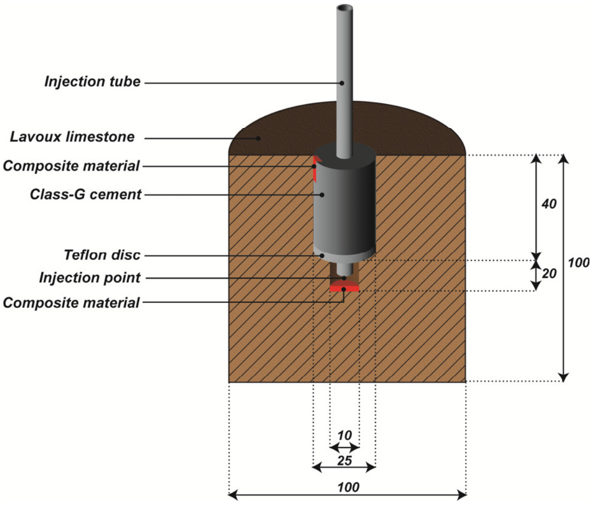


Fig. 1: Schematic view of the injection well of the MIRAGES-2 experiment. Dimensions are given in millimeters.

2.2. Description of the MIRAGES-2 experiment

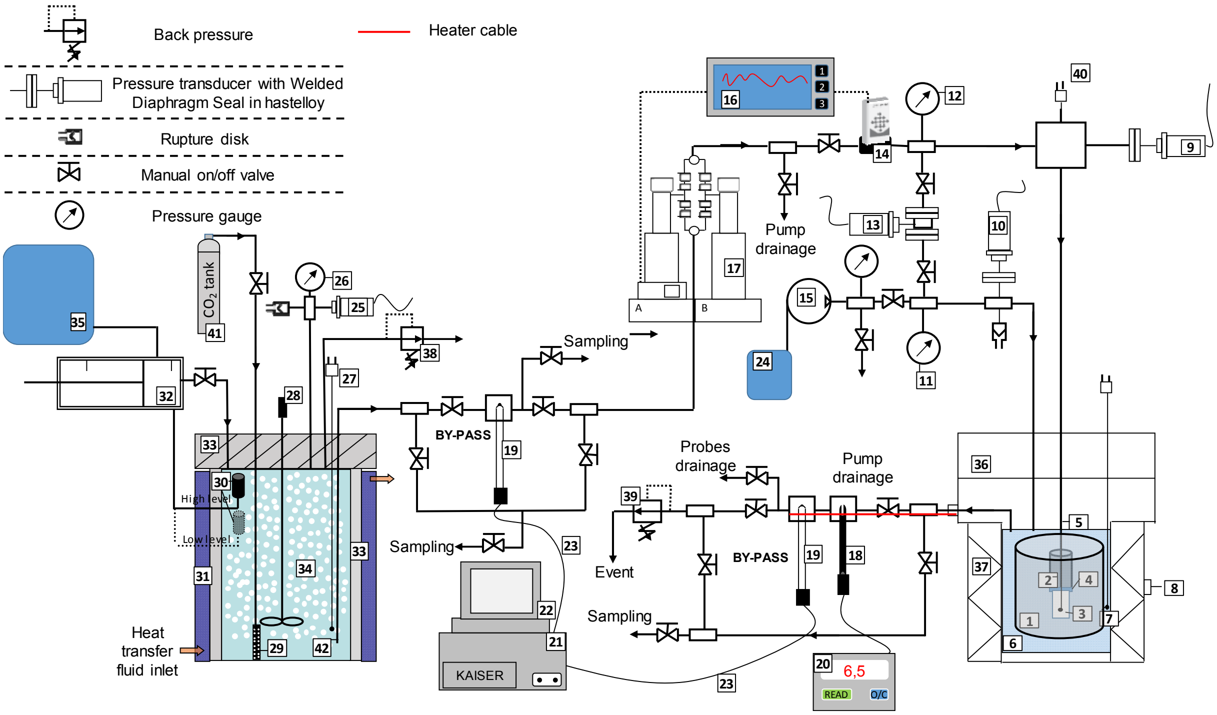
The experimental device is divided in two parts, the first one is devoted to the CO_2-solution mixing process, and the second one enables to perform the injection of the CO_2-rich solution in the core sample through the injection well, under controlled pressure and temperature conditions.

- Device for the CO_2/solution mixture

This device aims at preparing the appropriate CO_2 / H_2O mixing in order to obtain a monophasic aqueous solution containing the desired quantity of dissolved CO_2.

The mixing vessel is a 5 L autoclave made of stainless steel (316L-X2CrNiMo 17-12-2). The reactor walls and the cover are coated with a triple layer of Teflon (Fig. 2, item 33) to avoid corrosion problems due to the CO_2/brine solution. The body of the autoclave is surrounded by water-cooled temperature jackets to maintain the mixture at the desired temperature. The system is filled with water or brine with a displacement pump (10 cc) (Fig. 2, item 32) controlled by a water level sensor (Fig. 2, item 30) linked to a control unit station. This pump is machined in A-236 alloy (X6NiCrTiMoVB25-15-2). The nickel and chromium contents of this metal provide resistance to corrosion and oxidation. The CO_2 comes from a pressurized bottle (Fig. 2, item 41). The CO_2 goes through a 5 µm sintered (Fig. 2, item 29) and the solubilization is optimized with a static stirrer (Fig. 2, item 28). In order to prevent overpressure during the reinjection of the solution by the displacement pump, a back pressure regulator (Fig. 2, item 37) controls the pressure in the autoclave. The entire system can withstand up to 60 bar at 150°C.

When the CO_2-brine equilibrium is reached the solution exits from the bottom part of the autoclave using a dip tube (Fig. 2, item 41). A by-pass system is placed at the outlet of the tube to enable analysis of the mixture. It consists of i) a Raman probe (Fig. 2, item 19) connected to the system via a metal tee connector measuring the CO_2 concentration in the solution without any sampling; ii) valves for sampling the solution to perform chemical analyses or solubility measurement of the aqueous solution. At the output of the by-pass, the water with dissolved CO_2 fills a high pressure double syringe pump (Fig. 2, item 17) devoted to the mixture injection in the second autoclave containing the core sample and the well. This second autoclave is named the MIRAGES-2 reactor that is described in the next paragraph.



1	Core Sample
2	Cement
3	Injection zone
4	Teflon washer
5	Injection tube
6	Aqeuous solution
7	Confinement zone thermocouple (injection autoclave)
8	Oven Thermocouple
9	Injection pressure transducer (injection autoclave)
10	Confinement pressure transducer (injection autoclave)
11	Confinement pressure gauge (injection autoclave)
12	Injection pressure gauge (injection autoclave)
13	Differential pressure measuring transducer (injection autoclave)
14	Mass flowmeter
15	Hydropneumatic pump for water
16	Control unit of the pump controlled by the flow-meter
17	Syringue pump
18	In situ pH probe
19	In situ Raman probe
20	pH meter
21	Raman spectrometer
22	Raman data acquisition station
23	Optic fiber
24	Water tank
25	Mixing autoclave pressure transducer
26	Mixing autoclave pressure gauge
27	Confinement thermocouple of the mixing autoclave
28	Static stirrer
29	5 µm sintered
30	Level sensor
31	Heating jacket
32	Syringe for brine pressurisation
33	Mixing autoclave
34	CO_2/aqeuous solution mixture
35	Salted water inlet pré-balanced and filtered tank
36	Injection Autoclave
37	MICA heater band
38	Back pressure regulator for CO_2 (mixing autoclave)
39	Back pressure for CO_2/brine mixture (injection autoclave)
40	Injection zone thermocouple (injection autoclave)
41	CO_2 tank
42	Dipe tube for mixture collection

Fig. 2: Schematics of the MIRAGES-2 core flow experiment

- Device for the CO_2/solution injection (MIRAGES-2)

The experimental device MIRAGES-2 is based on a 2 L autoclave (Fig. 2, item 36) made of stainless steel (316L-X2CrNiMo 17-12-2) with a triple Teflon coating on the wall of the vessel and on the cover lid. This setup enables to inject the aqueous solution containing dissolved CO_2 through a core plug immersed in an aqueous solution.

Injection will be performed at constant flow rate using a double syringe pump (Teledyne Isco, Model 500D, Fig. 2, item 17) coupled to a liquid mass Coriolis flow meter (Fig. 2, item 14) via a control unit (Fig. 2, item 16).

This coupling allows working with injection pressures from 0 to 200 bar, with a flow rate ranging between 0.01 and 500 g/L (accuracy of 0.01 g/l). Due to the expected high chemical reactivity of the CO_2-brine mixture, the pumps are made of durable and corrosion resistant material (NITRONIC 50). To perform the injection in the core plug, a tube, either metallic or non-metallic (Fig. 2, item 5), is screwed onto the lower part of the lid of the 2 L autoclave. The confinement pressure is monitored by a hastelloy back pressure regulator (Fig. 2, item 39) and the temperature in the autoclave is reached using a MICA heater band (Fig. 2, item 37).

At the outlet, the fluid flows through a by-pass system equipped with high pressure and temperature probes to acquire pH and Raman spectra (Fig. 2, items 18 and 19). The probes used for in situ analyses materials are heated with a heater cable in order to keep a constant temperature in the analysis zone. The liquid sampling for chemical analyses is also possible thanks to a sampling valve located in the by-pass. The use of a bypass system makes possible the regeneration of the pH probe and a regular calibration.

The autoclave can also be pressurized by water before injection using a hydro-pneumatic pump (Fig.2, item 15).

High pressure rated check valves are introduced at strategic points in the system to avoid reverse flow in case of momentary pressure differences or pressure drops, preventing harmful damage to the system equipment. Moreover, rupture disk are also introduced in the two parts of the system to avoid any over pressure.

- Monitoring of the experiment

The two autoclaves of the experimental device are equipped with electronic sensors for pressure and temperature measurements and recordings. In the MIRAGES-2 injection autoclave, the pressure is measured and recorded at the injection point (Fig. 2, item 9) and in the confining zone (Fig. 2, item 10) with two high accuracy hastelloy flush diagram pressure transducers. To ensure accurate differential pressure measurements between the inlet and the outlet of the core plug, a hastelloy flush diagram differential pressure transducer (Fig. 2, item 13) is placed between the two zones.

During the experiment, regular measurements of the pH and of the dissolved CO_2 species are carried out with in situ probes. A regular calibration of the probes is performed before, during, and after the experiment to improve the accuracy of the measurements.

2.3. Experimental protocol

The syringe pump is used to inject continuously the CO_2-rich acidified brine from the mixing autoclave to the center of the core plug placed into the 2 L injection autoclave MIRAGES-2. The injection flow rate is set to 150 gram per hour (g/h). The temperature of the system is set to 60 °C and the confining pressure to 120 bar by adjusting the set point of the back pressure regulator. These P–T conditions are comparable with those of the geothermal fields currently exploited in the Dogger aquifer of the Paris basin. The entire duration of the experiment (15 days) was determined based on pre-dimensioning simulations carried out with a BRGM's in-house Thermo-Hydro-Chemical code.

Because the injected phase has to remain monophasic in each part of the experimental device, the CO2 concentration in the injected solution is always kept below maximum solubility value. The CO_2 solubility both in the mixture and in the MIRAGES-2 autoclaves were calculated using the thermodynamic model of Duan for the given pressure, temperature, and salinity [12]. In the reservoir condition (60°C, 120 bar), the solubility of CO_2 in pure water is 1.12 mol/kg. As a consequence, a pressure of 30 bar and a temperature of 20°C are imposed in the mixture autoclave to reach a theoretical solubility of 0.93 mol/kg, well below that calculated in the MIRAGES-2 autoclave conditions. This concentration prevents from any CO_2 exsolution in the MIRAGES-2 autoclave. The concentration of aqueous CO_2 is verified by Raman measurements in order to ensure that the system has reached the expected equilibrium concentration.

During the experiment the solution is sampled at the outlet of the two autoclaves to measure the quantity of CO_2 dissolved in water and to perform possible chemical analysis on major elements. The quantity of total carbonates is then determined by a $BaCl_2$ dosage under basic conditions. The pH of the output solution after having circulated in the core plug is also measured with in situ probe.

2.4. Analytical methods

- Raman spectroscopy

CO_2 concentration in the aqueous solution is measured with a Kaiser RXN1 Raman spectrometer. This is a portable compact system (Fig. 2, item 21) connected to a Raman optic head with an optical fiber (Fig. 2, item 23). This Raman head is connected to the in situ Raman probe (Fig. 2, item 19) which consists in a hastelloy tube composed of an optic lens and a sapphire window. The laser beam passes through the optical fiber, the probe and the sapphire window before interacting with the sample. The Raman signal generated by the light-matter interaction is sent to the spectrometer by the optical fiber. The peaks used in the determination of the solubility of CO_2 in water (Fig. 3) are those of the water bending vibration mode (~ 1640 cm^{-1}) and the CO_2 stretching vibration mode (~1340 cm^{-1}). The Raman shift ranges to measure the peak areas are 1500-1791 cm^{-1} for water and 1200-1450 cm^{-1} for CO_2.

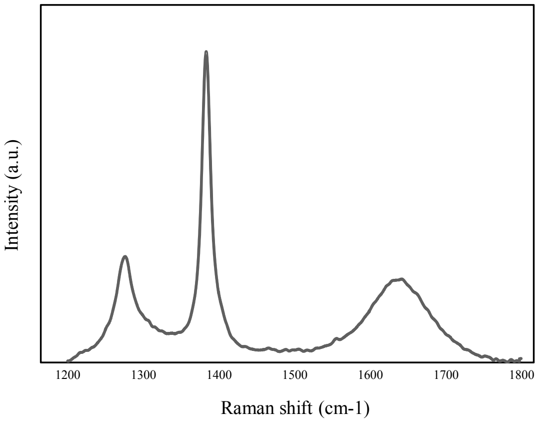


Fig. 3: Typical Raman spectra acquired CO_2 dissolved in water with the in situ Raman probes

- X-ray tomography

The X-ray tomograph is a Nanotom Phoenix (GE). This tool allows the exploration of the architecture of the solid samples with a resolution that can be less than one micrometer. The principle is to illuminate the sample with an X-ray beam and recording, using a photon detector X, the absorbed beam having passed through the solid to be analyzed. Images acquisition is repeated under different angles of rotation. The obtained sections are then reconstructed using the appropriate algorithms forming a three-dimensional image of the sample. This technique provides a non-destructive study that reveals the characteristics of the internal structure of the sample: size, shape, spatial distribution of the elements relative to each other, heterogeneities and defects (pores, inclusions, mineral phases ...).

3. Results of the first validation experiment

3.1. High pressure Raman probe calibration

The calibration of the Raman probe is carried out by measuring the area pic ratio of CO_2/H_2O (A_{CO2}/A_{H2O}) at 20°C (temperature of the solubilization experiment) for different pressures. The acquisition begins once the CO_2 is injected in the mixing autoclave. A sill assumed to correspond to the solubilisation equilibrium is reached after about 5 hours of CO_2 injection (Fig. 4, A). The calibration curve (Fig.4, B) corresponds to the linear trend fitted from the peak areas ratio A_{CO2}/A_{H2O} plotted against the CO_2 solubility calculated using the thermodynamic model of

Duan [12]. The correlation coefficient above 0.97 validates i) the calibration process and ii) the mixing process since the wanted amount of dissolved CO_2 is indeed present in the system.

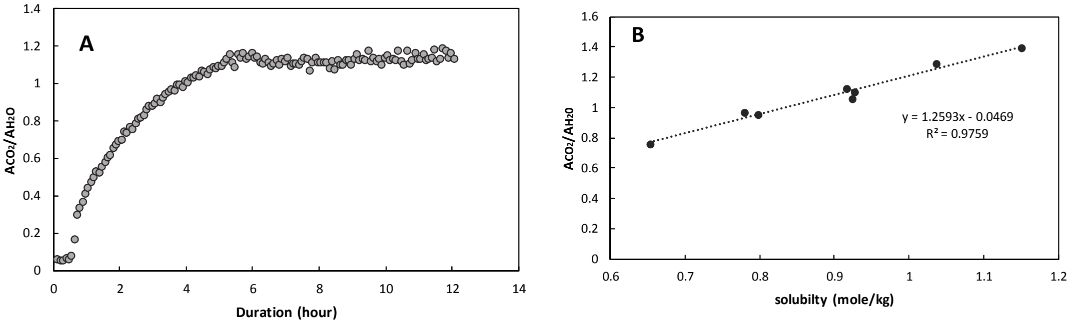


Fig. 4: (A) Evolution of the CO_2/H_2O area pic ratio (Raman analysis) during the solubilization of the CO_2 in water (P = 30 bar / T = 20°C); (B) CO_2/H_2O area pic ratio as a function of CO_2 solubility calculated from the Duan model at 20°C and different pressures.

3.2. High pressure pH probe calibration

The calibration of pH is performed before and after experiments to keep a good accuracy on the pH determination. The performance of a pH sensor generally degrades with time. This calibration allows to check the slope between the electrode potential and the effective pH. This slope defines the ability of the measuring electrode to change its output by 59.16 mV per pH unit at 25°C (~ 65mV per pH at 60°C) [13]. Fig. 5 shows that the potential electrode gives a linear response as a function of pH and that this slope does not evolve with time. The slope of about 64 mV per pH unit is in agreement with the literature data. It is of note that the second calibration curve with only two points is just used to check that the value of the slope has not changed during the experiment.

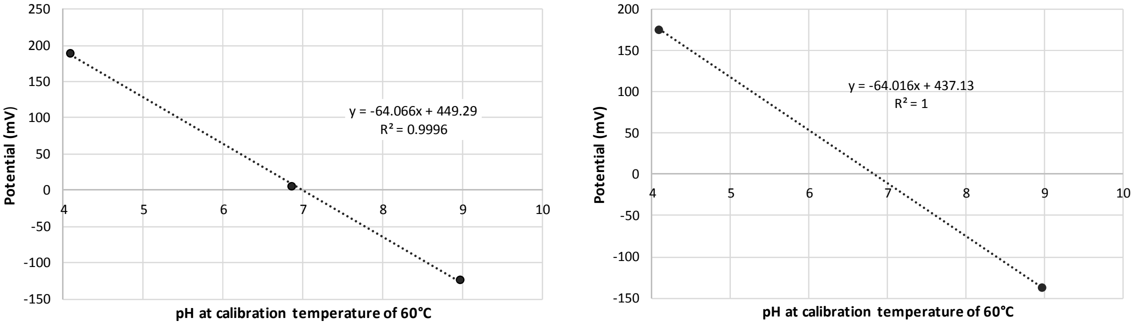


Fig. 5: Calibration curves of high pressure pH probe showing the electrode potential as a function of pH for two different calibration steps, before and after the end of experiment.

3.3. Monitoring of the first flow through experiment

After the steps of calibration of the different sensors and probes, a first experiment was performed with a core plug of Lavoux limestone. The injection tube is made of stainless steel sealed in the injection well with the class G Portland cement. This experiment lasted 15 days and was devoted mainly to the setting of the different parameters.

Fig. 6 shows the recording of pressures and temperatures in the mixing autoclave. The temperature remains stable all along the experiment with a mean value close to 20°C. The pressure is also stable around 30 bar with some variations due to the steps of filling of water or pumping the solution towards the injection pumps. The stability of these parameters guarantees a constant CO_2 concentration in the injected solution.

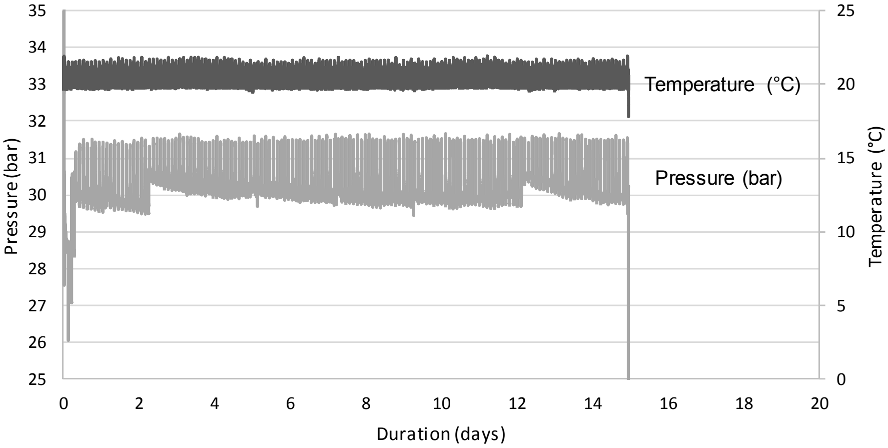


Fig. 6: Evolution of pressure and temperature in the mixing autoclave during experiment

The evolution of the confining and injection pressures and temperatures during the experiment are given in Fig. 7 and 8. It has to be noticed that the temperatures are well regulated and that the difference between injection and confining temperatures are around 0.5°C only, which is within the accuracy limits of the thermocouples. Concerning the pressures, they are relatively constant around 121 bar with some light variations attributed to the sensibility of the back pressure used to regulate the pressure in the MIRAGES-2 autoclave. The variations of the pressure gradient (ΔP) during experiment can be linked to the global variations of the pressure in the autoclave as well as changes of the permeability of the core plug.

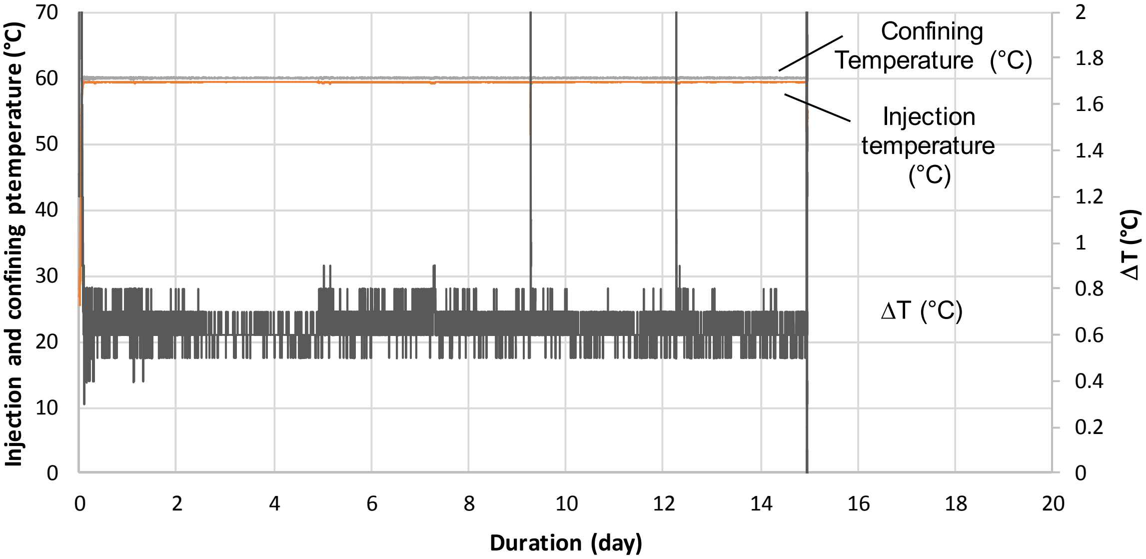


Fig. 7: Evolution of confining and injection temperatures in the MIRAGES-2 autoclave during experiment. The difference between the two temperatures is expressed as ΔT.

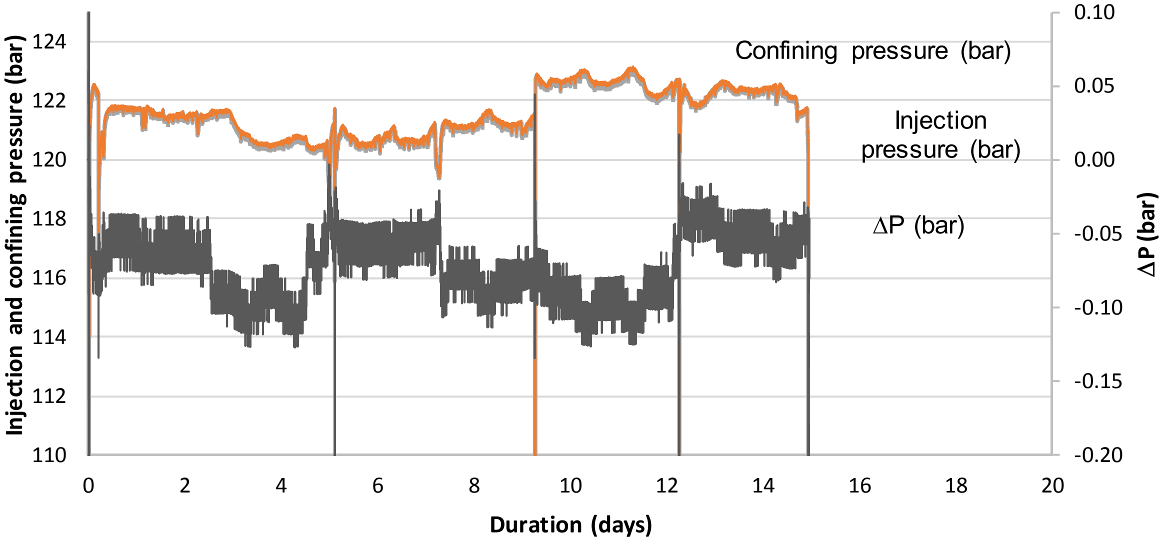


Fig. 8: Evolution of confining and injection pressures in the MIRAGES-2 autoclave during experiment. The difference between the two pressures is expressed as ΔP.

The flow rate of the injected CO_2-water mixture is regulated with a mass flow meter controlling the injection pumps. Fig. 9 shows the evolution of the mass flow rate with time. The injection is constant around 150 g/h with very punctual variations due to the steps of pump filling or to the action of the back pressure implying variations of pressure in the system.

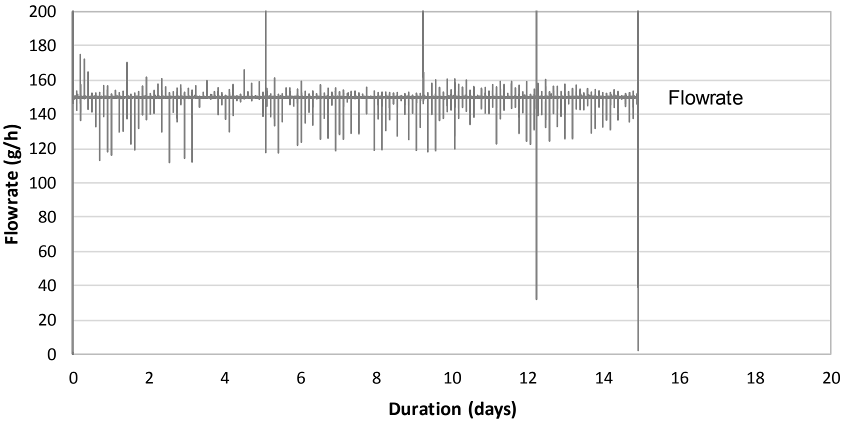


Fig. 9: Evolution of the mass flow rate injected in the core plug in the MIRAGES-2 autoclave during experiment.

The chemical parameters such as pH and dissolved CO_2 can be followed during experiment. Fig. 10 gives the evolution of pH directly measured under high pressure in the outlet solution at 60°C and 120 bar. One can notice that the solution tends to acidify with time showing that the chemical contribution of the limestone and of the cement evolves with time. The evolution of the concentrations of dissolved CO_2 is plotted in Fig. 11. During experiment, the CO_2 concentration in the outlet solution remains relatively constant with a value close to 1 mol/kg. A more precise analysis shows however a weak decrease of the CO_2 concentration at the end of experiment revealing a possible increase of the CO_2 consumption, possibly by the cement phases. This should be confirmed both by the analysis of the core plug and by the geochemical simulations.

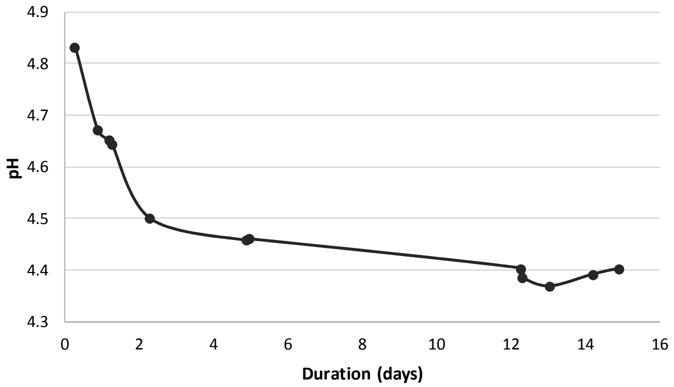


Fig. 10: Evolution of the pH measured in the outlet solution with in situ high pressure probe during experiment.

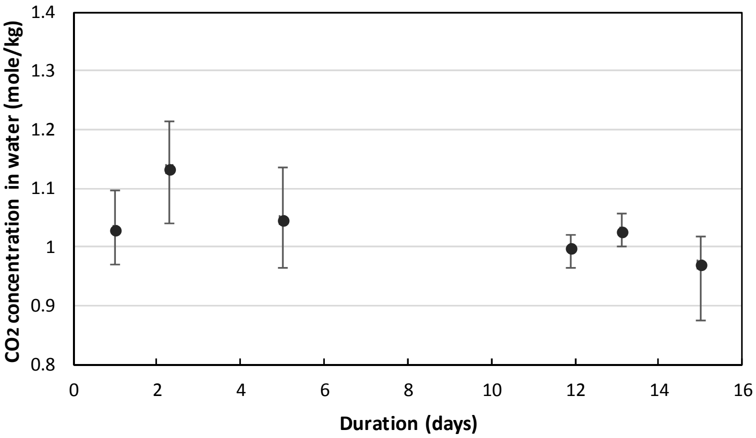


Fig. 11: Evolution of the CO_2 concentration in the aqueous solution measured with in situ high pressure Raman probe during experiment.

After experiment, the core plug shows different holes visible on the wall of the core plug (Fig. 12). The injection of the CO_2-water mixture has caused the dissolution of the limestone from the injection point to the exterior of the sample. X-ray tomography allows to visualize and reconstruct the porous network formed during this 15 days experiment. Fig. 12 shows a complex network with the formation of wormholing phenomenon which strongly affects the permeability of the system. This anisotropic pattern of the dissolution pathways observed here, while the injection process is basically isotropic from the bottom hole, strongly suggests the presence of an initial anisotropy of the porous network, possibly induced by the mechanical consequences of the sample preparation procedure. This will be assessed in the next series of experiment by imaging the sample both before and after the end of the experiment.

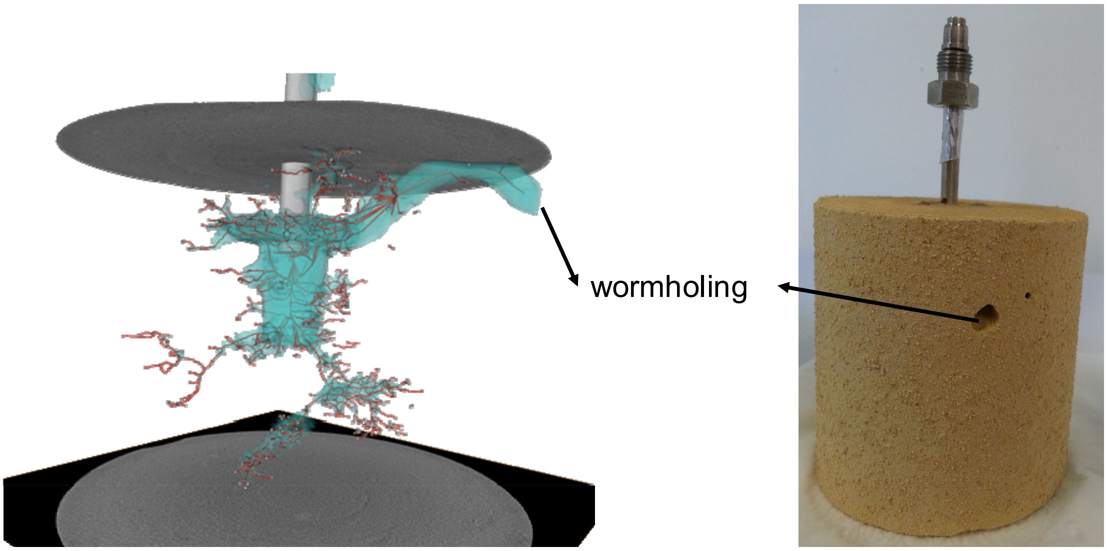


Fig. 12: Core plug after experiment. Left side: X-ray tomography image showing the porous network. Right side: the core plug after experiment showing the wormholing phenomenon.

4. Discussion and conclusion

The MIRAGES-2 experiment has allowed for the first time to mimic the injection of a CO_2-water mixture in a 1/20th scale model of an injection well. All the possible interfaces between materials (steel, cement, composite materials) and reservoir rocks (limestones and sandstones) can be studied with this experimental setup. The first results presented in this paper demonstrate the feasibility of such experiments and show that all the physico-chemical parameters such as pH, pressure, temperature, flow rate, dissolved CO_2 can be controlled during the experiment, even under high pressure conditions. This experiment which can be carried out with corrosive brines because of the inert material used can give some key points concerning the behavior of an injection well that undergoes the action of an aggressive solution. This MIRAGES-2 experimental setup has to be considered as an experimental model used to further validate the numerical simulation intended to be performed with coupled geochemistry / transport codes.

In the next steps of the CO2-DISSOLVED project, it is planned to test different materials for the well and the casing, other reservoir rocks (sandstone), and to quantify the chemical reactivity of each part of the injection well under the action of several saline CO_2-rich solutions.

Acknowledgements

This study was financially supported by the French National Research Agency (ANR).

Cedric Carpentier is thanked by the authors for his help to greatly improve the quality of the schematic view of the core plug.

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