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EXPERIMENTAL DEVICE TO EVALUATE MINERAL TRAPPING IN SANDSTONES AS A MEANS OF SUPERCRITICAL CO₂ (scCO₂) STORAGE

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ABSTRACT:

Carbon dioxide is the main Greenhouse Gas (GHG) due to its abundance in the atmosphere. CO₂ capture and storage (CCS) technologies contribute to mitigate the effects of climate change by reducing their emissions. This requires appropriate geological environments such as deep saline aquifers with great potential on an industrial scale in Spain. They must have appropriate characteristics to ensure the tightness of CO₂ and the technical viability of the injection process. The capacity of the storage is conditioned by the trap mechanisms that take place in depth. In this work, the static configuration of the ATAP (High Temperature-High Pressure) test device has been developed to reproduce the mineralization trap mechanism or mineral sequestration by applying it to a sandstone aquifer for supercritical CO₂ storage (SCCO₂). The equipment allows to observe the changes produced in a rock sample after it has been saturated with the fluids existing in the store (brine and SCCO₂), under the pressure and temperature conditions characteristic of storage (up to 120°C and 500 bar). To verify these changes, the technique of computed axial tomography and He pycnometry were used. The variations of porosity produced in the storage rock are compared with those obtained in studies of SCCO₂ saturation by injection and in reactor carried out by other authors.

Keywords: Mineral trapping, deep saline aquifer, sandstone storage, supercritical CO₂, high pressure and temperature equipment

Definitions and terms

ATAP	Experimental device- high temperature- high pressure
Artifact	Distortion of the radiological image
CCS	Carbon capture and storage
Completion	Cementation works, among others, carried out in producing wells or oil / gas injectors
DICOM	Digital Imaging and Communication on Medicine
XRD	X-ray diffraction
FOV	Field Of View
SCCO ₂	CO ₂ in supercritical state
SEM	Scanning Electron Microscopy
SSQD	Slow Suction Quick Delivery
Sw	Brine saturation
Sw _(100%)	Brine saturation at 100%
Swi	Irreducible brine saturation
CT	Computerized Tomography
Vp	Pore Volume
W	Brine

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1.- INTRODUCTION

Carbon capture and storage technologies (CCS) are presented as an option that could be viable to mitigate the greenhouse gas emissions more abundant in the atmosphere, constituting an alternative to fight against the climate change already proven [1]. The last stage addressed by these technologies is the geological storage of CO₂, being necessary to find suitable geological environments to ensure its success. Possible geological structures are the depleted hydrocarbon reservoirs, the underground coal layers and the deep saline aquifers [2], being this last option the one with most potential at industrial scale in Spain. Due to the need to find geological storages that guarantee the technical viability of the injection process and gas tightness [3], it is necessary to develop technologies capable of facing up these challenges associated with CCS projects.

The underground petrophysical and geomechanical properties have influence in the selection of a deep saline aquifer for CO₂ storage, also the scope of the trap mechanisms developed over time determine the capacity of the storage and the technical feasibility of the injection process. In addition, the tightness of the CO₂ storage in the long term must be guaranteed at all times. The trap mechanisms for CO₂ that can be developed in a deep geological storage were defined in 2005 by the Intergovernmental Panel on Climate Change (IPCC) [4], the times of action of each one of them has been collected in a general way in Fig. 1, during and after the injection process.

Figure 1. Estimated time of action for the CO₂ trap mechanisms. Modified from IPCC 2005.

The trap mechanism of mineralization or mineral sequestration is based on the chemical reactions that are generated between the minerals of the storage rock and the CO₂/brine mixture. The mineral dissolution produces new species that fix the CO₂ and modify the porosity and permeability of the storage, altering the pH of the mixture CO₂-brine-storage rock. While mineral sequestration increases with time, its speed of action is conditioned by the particle size of the storage rock, and by the temperature and pressure of CO₂ injection.

The need to increase the temperature represents a high energy consumption that can be avoided by the use of bacteria and fungi [5]. These produce the dissolution of organic acids creating protons that attack the mineral surface by the oxygenated parts, making the metal-oxygen barrier weaker, accelerating the dissolution of the magnesium silicates.

Seifritz [6] proposed in 1990 to apply carbonation reactions for the CO₂ storage of, being the first studies developed at Los Alamos National Laboratory (LANL) between 1995 and 1997 [7]. The aqueous carbonation consists on the dissolution of the CO₂ in the brine, which resulted moderately acidic. Subsequently, the Ca/Mg lixiviation occurs, ultimately precipitating the carbonates. The effectiveness of the mineral carbonation process is highly influenced by the type of storage rock (its mineralogy) and the methods or characteristics of the injection. Mineral carbonation could be improved if fully dissolved CO₂ is injected into water and/or if it is injected into silicates rich in divalent metal cations, such as basaltic and ultramafic rocks.

The mineral carbonation produced by CO₂ injected is different in extrusive rocks (volcanic basalt type) or in sedimentary rocks, being in the latter case a slower process [8, 9]. The CarbFix project [10,11] was developed with the objective of optimizing the technological development costs for in-situ mineral carbonation in basalts (mostly tolelic with Olivino). The injection zone rocks located south of the Hellisheidi geothermal plant (southwest of Iceland), have an ultrabasic to basaltic composition (with 45-49% SiO₂) with vitreous to crystalline structures. In the pilot phase, 175 tons of CO₂ were injected, previously dissolved in the brine, where the analysis of nine core feet confirmed that between 80-90% of CO₂ had produced carbonation phenomena in the rock in less than a year.

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In this work, the static configuration of a prototype at laboratory scale (ATAP) has been developed for the study of the mineralization trap mechanism or mineral sequestration produced in a sandstone aquifer for CO₂ storage. The equipment allows to observe the changes, reactions and precipitations produced in a rock sample, with the fluids present in the storage, in the real conditions of pressure and temperature characteristics of the storage. The variations suffered in the storage rock can be verified through complementary studies of X-Ray Diffraction, pH measurements, porosity and permeability measurements, or computed tomography with the sample located in the injection cell.

2.- MATERIALS AND METHODS

For the study of the trap mechanisms and the technical feasibility of CO₂ injection in a deep geological storage, a test equipment, ATAP (High Temperature-High Pressure), has been designed with the capacity to reproduce the actual storage conditions of up to 120°C and 500 bar. Through its dynamic configuration, studies have been made of physical CO₂ trap mechanisms developed in sandstone aquifers [12], phenomena of degradation in cements for the completion of injection wells [13] and improvement of injectivity in the storage [14]. For the analysis of the long-term CO₂ seal carried out in this work, it has been necessary to develop a static configuration of the ATAP device (Fig. 2) that allows analyzing the chemical trap mechanism of mineralization or mineral sequestration.

The injection of CO₂/brine corrosive mixtures at high temperatures and pressures requires the use of Hastelloy-type materials with resistances superior to stainless steel. In addition, the use of the CT (Computerized Axial Tomography) technique to visualize the changes produced in the sample under study, maintaining the test pressure and temperature, requires a radiolucent material that prevents the generation of artificial images. This material is a carbon coating in the area of the injection cell where the sample is located that allows the analysis of porosity, saturation, fluid displacement, homogeneities and heterogeneities of the samples, as well as the changes produced by mineralization processes.

Figure 2. Diagram of the static configuration of the ATAP device. Cell and sample with heat-shrinkable plastic

The rock sample is located inside the body of the cell with a diameter of 3.8 cm and a length that can vary between 2 and 8 cm, allowing the coupling of shorter samples using adapters in the pistons that close the central body. For this study, three samples of sandstone of the same formation, with a length of 6.5 cm, 6.8 cm and 6.9 cm respectively, have been tested which have been covered with a thermo-shrinkable plastic to prevent the diffusion of the CO₂ through the Viton jacket.

The tested rock is subjected to a radial confining pressure by the pressure increase applied to the glycerin located in the cavity between the Viton jacket that surrounds the sample and the walls of the cell. By the subsequent coupling of the cell, where glycerin is also located, the axial confinement to the sample is provided. In this way, pressure gauges and safety valves are used to control the tri-axial confinement pressure (140 bar) (radial + axial) of the rock, similar to that which would be subjected in a deep saline aquifer.

In order to maintain the constant temperature of the sample, the cell is surrounded by a radiolucent Teflon jacket that allows images without artifacts to be obtained inside a scanner, being able to analyze the changes produced in the rock during the test. The temperature of the jacket is kept constant at 35 °C by the circulation of the silicone oil pumped, previously heated with a resistance in an external tank.

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The injection/recovery of one or two fluids can be done independently or mixed before crossing the sample thanks the distribution spiral located on the surface of the two pistons that close the cell, thus avoiding its stratified entry. In this static ATAP configuration, one of the two piston inlets for fluid injection is closed while the other is used to inject first brine and then supercritical CO₂ (SCCO₂). In this injection line there is a valve and a pressure gauge at the entrance of the cell that allows to keep watertight the volume of fluid that saturates the sample during the test at constant pressure.

By means of a back pressure device, with a valve that closes the production of fluids at the outlet, it is possible to inject CO₂ into the sample in the supercritical state (SCCO₂), maintaining a pressure setting of the outlet pressure gauge at 74.5 bar, which is transmitted to the entire fluid injection system. By opening/closing the valve in the filling line with N₂, it is possible to vary the pressure of that backpressure equipment to the desired one in the test.

The continuous injection of fluids is carried out separately, on the one hand the brine with a high precision ISCO series-D pump at a flow rate (up to 107 ml/min) or constant pressure (up to 517.1 bar). On the other hand, the liquid CO₂ from the bottle by means of a JASCO PU-2080 generator with cooling of its heads for the thermal control below -4°C, is pumped in supercritical state avoiding the phase change by decompression when entering the pump. The SSQD pump system provides flow stability (free of pulses) achieving a flow rate of up to 10 ml/min and a maximum pressure of 35 MPa.

The effect of the mineralization trap mechanism is analyzed before and after the injection test using the He pycnometry and the CT techniques (Somatom medical scanner from the Siemens Company). The images obtained from the sample are compared with a base image fixed for a reference situation in both the cell and the scanner table (Fig. 3).

Figure 3. Reference position of the cell on the scanner table.

The brine injection is carried out at a constant flow rate (1cc/min) until 20 times the pore volume of the sample is injected, at which time we consider it is 100% saturated in brine $S_{w(100\%)}$. Once the pressure and temperature conditions have been established inside the cell, with the sample brine saturated and the connection valves closed, the *study position* is set to obtain the sequence of CT1 images of the sample during the test by means of the scanner (Fig.4).

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Figure 4. Phases of the injection test and sequence of the computed tomography record CT1-3

The injection of $scCO_2$ is then carried out at a flow rate of 1cc/min, obtaining an equivalent volume of injection of 10 times the pore volume (Vp), displacing the brine from the sample inside. The injection continues until no more brine is produced and only CO₂ (g) is recovered at the outlet of the cell. At that time it is considered that the sample is saturated in $scCO_2$ and in the volume of undispersed brine that constitutes the irreducible saturation S_{wi} . The injection and production valves of the cell are closed and the second computed tomography image CT2 is obtained (Fig. 4). The cell is kept sealed at constant temperature and confining pressure for two months, during which the mineralization process takes place by reaction of the rock, brine and $scCO_2$ in the sample (mineralization trap mechanism). A third CT3 computed tomography study was performed with the cell in the *study position* (Fig. 4).

Once the CT3 study is done, valves are opened and the sample inside the cell is recovered. The sample is placed in an oven and kept at 25 °C until it is dry, then, its porosity is measured after the test in the He Pycnometry equipment.

3.- RESULTS AND DISCUSSION

The images obtained with the scanner by means of the field of view (FOV), in the selected window, are compared to observe the possible changes that affect the sample in the different stages of the test. By means of the software with image acquisition, with DICOM standard, the slices are obtained (approximately 107 per study) made in the saturated sample in brine and later in $scCO_2$, calculating the average intensities in Hounsfield units. The image composed of intensity measurements in gray scale shows variations from white to black that determine the density of the material in the different points crossed by the X-rays. The white corresponds to the densest material and the black to the least dense. Through the image treatment program, the maximum intensity measurement is obtained in each of the slices (Fig. 5).

Figure 5. Maximum intensity measurements of each of the slices in each stage

Figure 6 compares the images obtained from the first sample of sandstone (representative of the formation) inside the cell at the pressure and temperature of the storage in four slices at 6, 12, 18 and 24 mm distance from the injection surface, saturated in brine (CT1), the sample saturated in SCCO₂ and the irreducible volume of brine (CT2), and two months after the sample saturation with SCCO₂ (CT3). The comparison of the studies carried out in each of the stages and in the positions fixed in the sample is then carried out.

Figure 6. Scanogram of the sample located inside the cell. Images of the scanner in the different stages and study positions

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For the first sample of tested sandstone, no significant qualitative changes are observed as a result of the different injections. This is due to there is not variations of the macro porosity in the images provided of the samples by the CT technique.

After two months of residence of the SCCO₂, the cell is opened and the sample is recovered, which, once is dried in an oven, is subjected to a quantitative study with the He Pycnometer, obtaining a total porosity of 10.90%. Table I also shows the porosity results (9.42% and 8.85%) of the other two tests carried out in sandstone samples of the same formation, at 35°C of temperature and respective pressures of 140 bar and 75 bar.

In another study conducted by Campos et al [15] where a sandstone sample from Utrillas was subjected to the injection of SCCO₂ and brine under storage conditions (80 bar and 32°C), a decrease in its porosity was measured with Hg porosimetry, 15.64% to 9.70%, similar to that observed in the samples used in our study (Table I). This decrease of the meso porosity due to mineral precipitation leads to a lower storage capacity of the CO₂ injected later. Although this contradicts the studies of Berrezueta et al [16], where there is an increase in porosity, this is a phenomenon that depends not only on the mineralogy of the storage and the composition of the brine, but also on the time of CO₂ permanence in the aquifer.

Sample		Test			Porosity measurement			Ref.
Rock	Location	Device	P&T	Duration	Method	Before	After	
Sandstone	Duero Basin	Injection SCCO ₂ - W	140 bar 35°C	2 months	He Pycnometry	16,97	10,90	This work
			75 bar 35°C			14,85	9,42	
						12,53	8,85	
Sandstone	Ebro Basin	Injection SCCO ₂ - W	80 bar 32°C	2 months	Hg Porosimeter	15,64	9,70	Campos et al, 2015
Feldespar Sandstone	Guadalquivir Basin	Reactor with SCCO ₂	75 bar 35°C	1000 h	OpM and SEM	12,63	12,94	Berrezueta et al, 2017
Greywacke	Ebro Basin					19,01	21,16	
Quartz- sandstone	Duero Basin	Reactor with SCCO ₂ - W	75 bar 35°C	24 h		9,55	11,13	

SCCO₂: supercritical CO₂; W: brine

OpM: Optical image analysis with thin-section; SEM: scanning electron microscopy

Table I. Comparative results of SCCO₂ injection in different types of rock and test conditions

Table I shows the difference in the variation of the porosity according to the methodology used to lead the aquifer sample to the pressure and temperature of the storage conditions. An increase in porosity occurs when performing the tests in the reactor, both with the sample submerged in SCCO₂ and in the SCCO₂-brine mixture, compared to the decrease produced when the SCCO₂-brine mixture is injected into the sample (located inside the triaxial cell). Further complementary and sensitivity studies would be necessary to justify the variation of these results, taking into account the influence of factors such as the mineralogy of the sample, the composition of the brine and the technique used to introduce supercritical CO₂ into the rock.

After the SCCO₂ injection, its transformation into CO₂ (aq) and HCO₃⁻ is first produced by dissolving it in the brine, thereby increasing the safety of the store, since no CO₂ is found in the free phase. Subsequently, mineral dissolution is caused by varying the pH of the SCCO₂-brine mixture [17]. These processes produce a decrease in system pressure and the consequent separation of CO₂ from the aqueous phase as its solubility decreases, creating a CO₂-free phase again. All this causes the subsequent mineral precipitation, producing changes in the morphology, porosity and permeability of the porous medium, which then affect the natural biphasic flow of CO₂ and the formation water (brine) inside the storage, influencing the storage capacity and security, being necessary to study in each case those variations to know their contribution.

Additional studies could be carried out to obtain the pH of the effluents, the characterization of the mineralogy and petrography by means of XRD, SEM or optical image analysis [16,18,19], as well as petrophysics with the measure of the variation of the permeability. Likewise, the scope of the study of the characteristic chemical reactions of the

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mineralization trap mechanism could be broadened based on the geomechanics of the storage and the possible effects on the sealing of CO₂ in the aquifer [20].

4.- CONCLUSIONS

The ATAP equipment in its static configuration has allowed reproducing the chemical trap mechanism of mineralization developed by injecting CO₂ into the sample at the pressure and temperature of the deep saline aquifer. The test time of the sample under these conditions allows the study of the chemical reactions that occur between the rock, brine and SCCO₂ characteristics of the mineral sequestration.

The studies with CT of the inner part of the sample located in the cell (at the pressure and temperature of the test) make it possible to observe the variations in its mineralogy, and if fracture or cavity phenomena have been generated. It has been found that the variation in the porosity of the tested sandstone sample was not appreciable enough with this technique. Its application is reduced to cases in which significant qualitative changes occur, either by the mineralogy of the storage (basaltic rocks) where the mineralization processes are more urgent in the short term, or by more aggressive chemical reactions due to the composition of the brine.

For the complete study of mineral sequestration developed in underground storage, an assay methodology should be followed where, prior to the injection of the fluids of known composition, the mineralogy and porosity of the samples to be tested are analyzed. It is recommended to use analytical techniques such as the XRD and SEM in the injection surface, which allow to select the areas of interest, and by means of mercury porosimetry to quantify the pore sizes and volumes, as well as their distribution and interconnection.

The nature of the mineralization trap mechanism determines that the time of permanence of the CO₂ in the supercritical state in the cell during the test is a function of the mineralogy of the sample, and can be reduced with the use of catalysts that accelerate the chemical reactions. In this case, it is convenient to study the chemical reactions that may develop in the aquifer. It is suggested the possibility of measuring the pH of the initial and final effluents, after the permanence of the supercritical CO₂ in the sample and observe its variation. It would be convenient to analyze the variation of its mineralogy, in the same points prior to the injection, check the variation of its porous structure, and study the chemical composition of the effluents obtained.

The dissolution and mineral precipitation modify the porous structure expanding the throat diameters and decreasing the specific surface area of the pores, resulting in lower capillary pressures and changes in relative permeability. As a result, there is an increasing resistance to the flow of brine and a decrease in the mobility of the injected CO₂, acting as a self-sealing mechanism, being unfavorable to the migration of CO₂ to leakage areas (faults, fractures) or seal. The study with ATAP and the complementary tests of the processes developed in each storage, conditioned by its mineralogy and its specific geology, leads to a more precise knowledge of the storage capacity of the geological formation and of the safety in the sealing of the CO₂ in the storage in the long term.

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