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Evolution of the microstructural and chemical properties of the CO_2-aged Banco Verde sandstone

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ABSTRACT

With the exponential increase in the World's mean temperature, produced due to the worrying rise in CO_2 emissions, more and more international entities are looking for options to mitigate this natural phenomenon. In this context, researchers have proposed storing this gas in the subsoil. Among the different types of CO_2 underground storage, a commonly mentioned alternative is the use of depleted oil and gas reservoirs. These formations are characterized by being composed of sedimentary porous reservoir rocks, covered by a caprock layer that stands out due to its low permeability. In Argentina, three main basins are considered as possible CO_2 storages, being the Salamanca Formation from the Golfo San Jorge Basin the case study of this article. This formation is composed of three seal layers (Lignitífero, Fragmentosa, and Banco Negro) alternated with two reservoir rocks (Glauconítico and Banco Verde). It is of particular interest the study of the Banco Verde layer, as it is a depleted oil and gas reservoir that seems to gather the necessary properties to be used as a permanent underground CO_2 storage. In previous research, laboratory mechanical tests were performed on pristine and CO_2-aged sandstone samples, showing a notable variation in their properties after CO_2 exposure. Aiming for a better understanding of this property evolution, microstructural and chemical analysis such as X-ray diffraction, and Mercury Intrusion Porosimetry, were performed. The observed modifications in the behaviour of the reservoir rock seem to be consequent with the microstructural and chemical evolution produced.

KEYWORDS

Sandstone CO_2-aged; Carbon capture and storage; Depleted oil and gas reservoir; Micro structural and chemical property evolution.

1. INTRODUCTION

According to the Intergovernmental Panel on Climate Change, in the last 100 years, the average global temperature has increased by about 1.1ºC (Working Groups I, II and III of the Intergovernmental Panel on Climate Change (2023)). This notable increase in temperature leads to substantial changes in the daily activities of the species that inhabit the earth's surface. This is why the issue of climate change is constantly being addressed. Considering the fact that this phenomenon is mainly produced due to the exponential increase in the emission of greenhouse gases (Siddiqui et al. (2022)), 75% of which are carbon dioxide (CO_2) (Working Group III of the Intergovernmental Panel on Climate Change (2014)), researchers are currently looking for ways to adequately dispose these fluids.

Carbon dioxide capture and storage is a practice that is being increasingly mentioned, as it represents a viable alternative in the process of mitigating global warming. It is intended to reduce the concentration of CO_2 in the atmosphere through the geological storage of the gas in a suitable rock stratum. There are different types of geological formations whose geomechanical properties classify them as suitable to be considered as CO_2 reservoirs. These include coal beds, salt beds, depleted oil and gas reservoirs, and saline aquifers (Bachu et al. (2000)).

In 2015, in the context of the United Nations Framework Convention on Climate Change, the Secretariat of Environment and Sustainable Development of Argentina met with the aim of proposing measures to mitigate global warming. Thus, among the options mentioned, the geological storage of CO_2 was highlighted, proposing different depleted gas and oil formations as possible reservoirs of this greenhouse gas. Among them, particular interest was shown in the oil formations distributed in the Golfo San Jorge Basin. At that time, it was considered that the country did not have the resources or the necessary experience to start applying these practices, so the corresponding investigations were not carried out. Despite this, in recent years, several Argentinian researchers have shown interest in focusing on this research area (Manzanal et al. (2013), Barría et al. (2018), Martín et al. (2022), and Grasetti et al. (2022)). Except for particular cases (Grasetti et al. (2022)), the studies developed focus on the study of the interaction between the cement of the injection well and the injected CO_2 and on the modelling of the CO_2 interaction with the cement and not on the evolution of the properties of the rock in charge of storing the gas in question.

When considering the feasibility of using a certain rock as a CO_2 reservoir, it is essential to study the modifications produced in its properties due to the action of supercritical carbon dioxide ($scCO_2$). The geomechanical properties of interest depend mainly on two factors, on the one hand, to ensure that the gas is fixed (Bradshaw et al. (2007)), and on the other hand, to ensure the long-term integrity of the reservoir (Hawkes et al. (2004)). Thus, the main geomechanical properties analysed are usually porosity and permeability, composition and strength.

Considering the national interest in developing geological storage of carbon dioxide, and the scarce research developed on this subject, this article proposes the Banco Verde stratum of the Salamanca Formation, belonging to the Golfo San Jorge Basin, as a possible CO_2 store. The proposed stratum is formed by a glauconitic sandstone of marine origin, which is characterized by being a reservoir of depleted gas and oil (Foix (2009)). Thus, a detailed analysis of the compositional and micro structural properties of the chosen sandstone, before and after subjecting it to the action of $scCO_2$ for a period of 30 days, is presented below.

2. MATERIALS AND METHODS

2.1. Salamanca Formation description

According to Feruglio (1949), the Salamanca Formation is composed of five different layers. The lower layer, known as Lignitífero, composed of grey clays with thin sandy banks containing abundant carbonaceous remains, with a thickness varying between 15 and 20m (TERMAP S.A. (2011)). Directly in contact with this last is Glauconítico (10-30m), which, as its name indicates, is a glauconitic sandstone stratum with low clay content, being this the first oil and gas reservoir found and exploited from the Golfo San Jorge Basin. The Fragmentosa layer, is characterized for being the thickest of the stratum composing the Salamanca Formation (140m), representing the cap rock of the Glauconítico, being composed of a fractured grey claystone filled with sandy sediments from the above layer. The two strata left, Banco Verde and Banco Negro, from bottom to up, represent the shallower oil and gas reservoir of the Golfo San Jorge Basin and its corresponding cap rock. The special interest shown in them as possible geological storage of CO_2 resides in the fact that the Banco Verde reservoir rock is depleted. Additionally, this layer is composed of coarse to very fine-grained greenish-gray sandstone, material discontinuities such as gypsum veins, glauconite and vulcanite clasts and clay lenses that come from the Banco Negro stratum (Foix (2009)). According to Rodríguez et al. (2014), Banco Verde is characterized for having high values of porosity and permeability, which make it a suitable reservoir rock for CO_2 injection. The Banco Negro layer, represents the cap rock of the Banco Verde reservoir rock, adding safety to the possible underground CO_2 storage. This claystone is composed of dark gray clays, with black speckles, characterized for presenting low permeability values, which make it a potentially effective sealing rock for potential gas leaks.

The presented study aimed to analyze the variation produced in the properties of the Banco Verde reservoir rock layer due to its aging with scCO_2 during a 30-day period. To achieve this, it was first necessary to obtain samples from which to extract specimens to be further tested in both its pristine and carbonated state. Bearing in mind that it was economically unfeasible to obtain underground samples, the alternative chosen was to extract samples from outcrop. Even when Salamanca Formation can be reached superficially in several geological locations, such as "Estancia las Violetas", "Puerto Visser", "Meseta del Curioso", among others (Foix et al. (2009)), just in some of them, the Banco Verde layer is reachable, being this the reason why the "Punta Peligro Norte" outcrop was selected. In this location, Fragmentosa, Banco Verde and Banco Negro layers of the Salamanca Formation can be reached. With the help of a pickaxe, samples were extracted from the lower part of the Banco Verde sandstone and taken to the "Laboratorio de Investigación de Suelos, Hormigones y Asfaltos" of the Universidad Nacional de la Patagonia San Juan Bosco (UNPSJB) for further treatment.

2.2. Specimen obtention

Once the samples were obtained from outcrop, the next step was to extract and prepare the specimens to be tested. Bearing in mind that the specimens to be subjected to mineralogic and micro structural analysis had to be pieces of rock of around 50g, depending on the test, and with no particular shape, it was considered adequate to take advantage of the pieces that resulted from performing the mechanical tests.

The cores to be used for mechanical property determination were obtained according to the ASTM D 4543 – 01 normative, using a coring machine and a saw. Once these were measured and weighed, the specimens were paired according to their property similarity, considering them "twin" specimens. Half of the twin specimens were stored in a constant humidity and temperature chamber, while the other half were subjected to a scCO_2-aging process. The CO_2 was injected into the chosen sandstone specimens during a 30-day period, completed using a carbonation cell available in the UNPSJB's laboratory. The gas was characterized for having a pressure of approximately 10MPa and a temperature of 60ºC, conditions selected with the aim of injecting the CO_2 in a supercritical state of matter (according to Ranjith et al. (2013), CO_2 is supercritical at 7.38MPa and 31.48ºC) and reaching the real underground temperature of the reservoir. Both groups of specimens, pristine and carbonated, were subjected to the desired mechanical tests.

As it was previously mentioned, after performing the mechanical tests on the twin cores, the resting pieces of rock were used to obtain the necessary specimens to perform the mineralogic and micro structural analysis. Considering that the objective of the research was to study the variation of the chemical properties of the glauconitic sandstone due to its exposure to CO_2, pieces of rock were extracted from the already tested cores from two zones: centre and edge. In this way it would also be possible to view the degree of penetration of CO_2 in the carbonation process, bearing in mind that the edge of the cores would be exposed to the gas from the first moment, while the centre wouldn't.

2.3. Laboratory tests

Previously, it was pointed out that when studying the feasibility of using a certain geological stratum as a storage for CO_2, it was necessary to consider, on the one hand, the available volume and, on the other hand, the type of trapping that occurs (Bradshaw et al. 2007). Thus, the need arose to study the evolution of the microstructural properties of the sandstone that forms the Banco Verde after CO_2 injection. This analysis also allows a better understanding of the modifications presented in terms of the mechanical properties of the rock. In this context, it was proposed to study the evolution of the rock composition, as well as its porosity and pore distribution.

In order to analyse the variation of the aforementioned microstructural properties, two different tests were carried out, X-ray diffraction (XRD) using the Panalytical X'Pert PRO X-ray diffractometer equipment and Mercury Intrusion Porosimetry (MIP) using the Micromeritics 9500 AutoPore IV equipment, both available at the Laboratorio de Geotecnia - CEDEX. These were carried out using pieces extracted from both groups of specimens, exposed and unexposed to the action of scCO_2, previously subjected to mechanical tests. Additionally, to complement the compositional tests, thin sections were obtained from the pristine sandstone.

3. RESULTS AND DISCUSSION

3.1. Porosity and pore size distribution

When performing the MIP tests, two general aspects of the specimens studied can be analysed. On the one hand, the porosity variation in terms of the pore diameter can be determined. From here, not only the porosity to mercury intrusion of the specimen can be determined, but also, a certain idea of the pore size distribution can be reached. Both analyses can be developed based on a porosity vs. pore diameter plot. On the other hand, by representing the differential intrusion in terms of the pore diameter, a more detailed analysis of the pore size distribution present in the specimen tested can be obtained.

In Figure 1.a, the porosity of the tested specimens is plotted in terms of the pore diameter. The curves represent a mean curve obtained from averaging the results derived from performing several tests for each type of specimen tested (5 pristine sandstone specimens, 3 30-day carbonated sandstone specimens from the centre and 3 from the edge). Three main observations can be made from these curves. Firstly, it can be observed that there is a reduction in the porosity of the sample, varying from 10.65% for the pristine sandstone to 10.34% for the 30-day carbonated sandstone from the centre of the core and 10.08%. Secondly, the amount of mercury that remains inside the specimen once the extrusion process is completed, reduces in a higher rate than the porosity of the sample, starting at a value of 5.93% for the pristine sandstone and decreasing to 4.53% y 3.49% for the CO_2-aged specimens from the centre and edge respectively. According to Cebeci et al. (1978), this remaining or entrapped mercury, is related to the ink-bottle pores (pores connected to a narrower-neck capillary) present in the specimen. Bearing this in mind, it seems reasonable to think that the CO_2 injection produces a reduction in the specimen's ink-bottle pore content. Finally, from comparing the shape of the curves, it might be noted that there is an important change produced in the pores with a diameter varying between 100nm and 10000nm. For a better understanding of this last, Figure 1.b should be analysed in detail.

When studying the pore size distribution that characterizes the pristine glauconitic sandstone specimens, it might be mentioned that it seems to have pores of two main sizes: 40nm-90nm and 800nm-4000nm. These pore size distribution might be observed in Figure 1.b, noting that for the 30-day carbonated specimen from the centre, the two groups of pore sizes continue to predominate, while in the case of the altered sandstone specimens obtained from the edges of the core, the pores corresponding to the 800nm-4000nm group, seem to have suffered an important reduction, while the pores with a size between 40nm and 90 nm, seem to have increased in amount. Similar changes in pore distribution were reported by Zheng et al. (2015), presenting a reduction in porosity as well. As it was previously mentioned, from observing this figure together with Figure 1.a, to understand why the change in the plotted in porosity vs. pore diameter have a different shape for each specimen type.

The variations produced in terms of the porosity and pore distribution of the Banco Verde sandstone after exposing it to a CO_2-rich environment can be summarized as follows. In the first place, as Laskowski et al. (2023) had shown for different samples obtained from outcrop, the glauconitic sandstone studied seems to always present a reduction in its porosity. Additionally, there could be a reduction in the amount of ink-bottle type of pores, noted from comparing the entrapped mercury at the end of each of the tests performed for each type of specimen, which could also be related to the reduction in bigger pores and the increment in smaller pores. Finally, it has been made evident from comparing the three types of specimens, that the centre of the carbonated core is less affected by CO_2 than the edge. This is reasonable to think as the exposure time of the outer part of the core is longer, given the fact that this zone is in contact with CO_2 from the first moment while the inner part becomes exposed when the diffusion process is completed and reaches the centre.

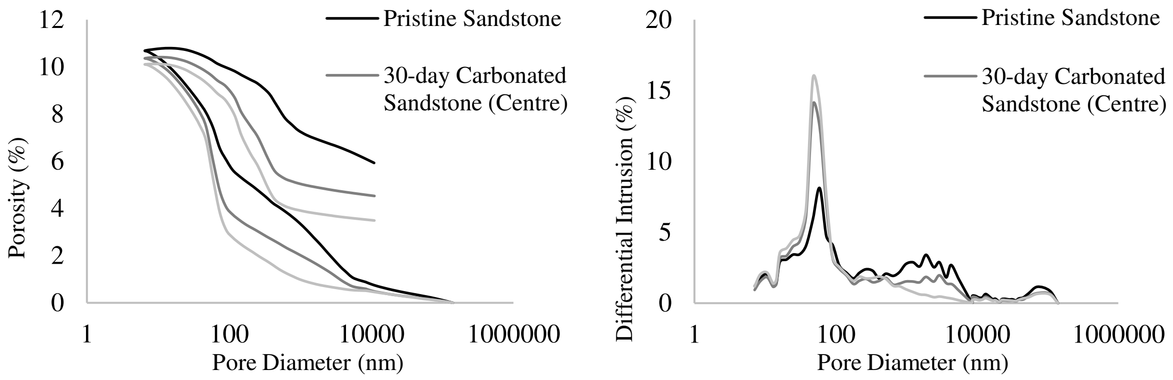

Figure 1.a) Porosity vs. Pore Diameter plot. b) Differential Intrusion vs. Pore Diameter. Plots obtained from performing MIP tests on pristine and carbonated specimens, from both edge and centre zones.

3.2. Element composition and mineralogy

With the aim of analysing the variation produced in the composition of the sandstone due to its exposure to a CO_2-rich environment, X-ray diffraction (XRD) tests were performed in pristine, and 30-day carbonated specimens previously subjected to mechanical testing. In particular, when testing the altered rock, pieces were extracted from the edge and centre of the core, in order to analyse the penetration degree of the CO_2 in the core during the carbonation process. To complement the XRD, the petrography of thin sections of pristine sandstone was studied.

In general terms, sandstones are characterized for being composed of quartz, feldspar, mica, and rock fragments, among other components (Davies et al. (1975)). When analysing the petrography of the Banco Verde sandstone (Figure 2), the prevailing mineral was the quartz, representing around a 30% of the total composition. Additionally, feldspar (~10%), glauconite (~10%), carbonates (~5%) and volcanic rock fragments (~5%) were detected. From this analysis, it was also noted that the rock matrix was composed of calcite cement, presenting in some grain contacts, dolomite crystals and clay (Figure 2.b). From observing various thin sections of the pristine sandstone in the microscope, it could be concluded that the sandstone layer studied had also plagioclase grains (type of feldspar) throughout its volume.

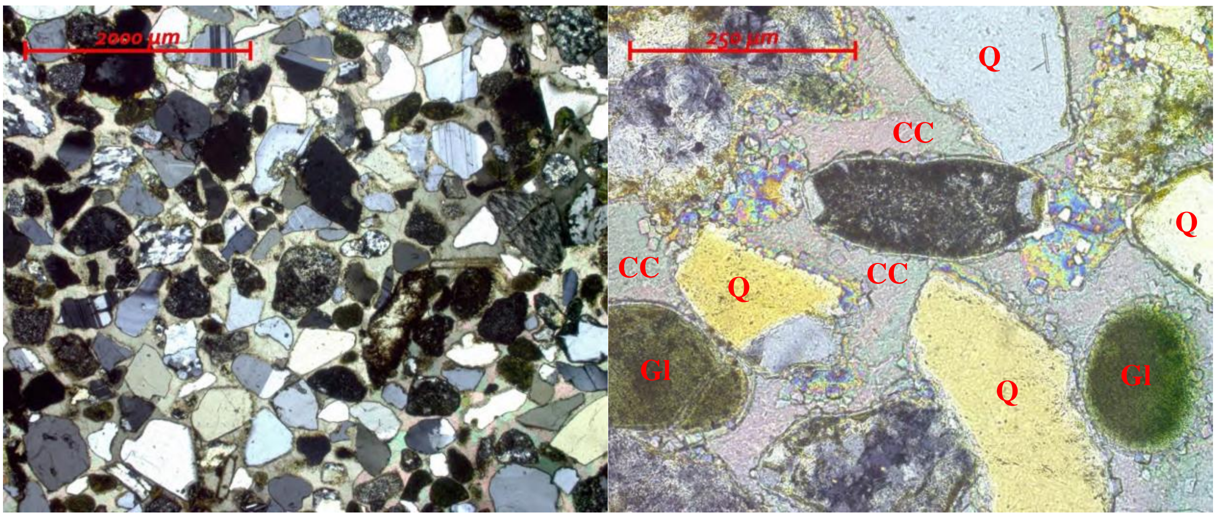

Figure 2.a) General aspect of the thin section of pristine sandstone under analyzed or cross polarized light. b) Zoom into the thin section of the pristine sandstone under analyzed or cross polarized light. Q: quartz, Gl: glauconite, CC: calcite cement.

All the aforementioned minerals composing the Banco Verde sandstone, were also present in the XRD tests. As it can be observed in Figure 3, the quartz content is approximately 30%, as indicated by the petrographic analysis of the thin sections, and there is presence of plagioclase, indicated by the appearance of albite and anorthite. When studying the variations produced in the sandstone due to its exposure to a CO_2-rich environment, four main changes seem to occur in compositional terms. One of the most notable changes is that quartz content seems to be increasing. It can also be observed that there are changes in percentage of albite and anorthite present in the sandstone after its exposure, indicating that plagioclase is being altered by CO_2. Finally, there is a reduction in calcite content, mineral principally composing the cement matrix of the Banco Verde sandstone.

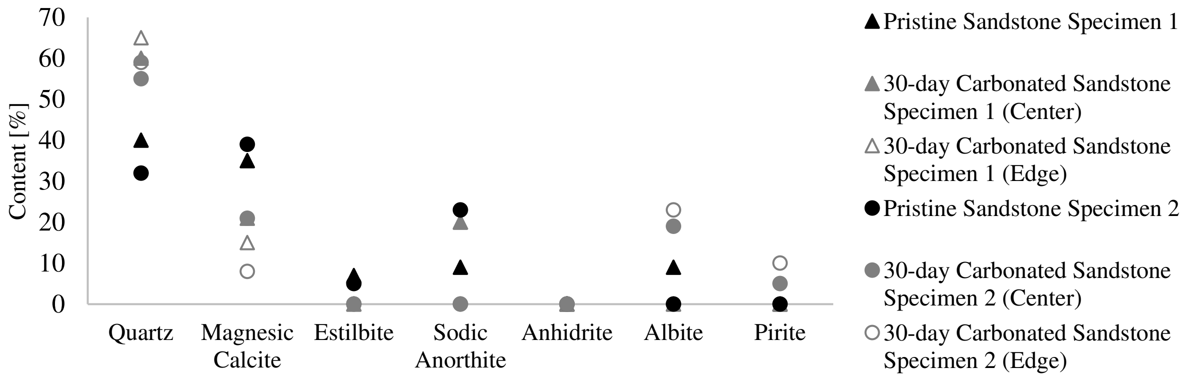

Figure 3. Mineralogic content determined through XRD tests over two groups of twin specimens.

Before analysing the mentioned changes in quartz and plagioclase, two aspects should be considered. On the one hand, a reduction in the core's weight after being exposed to CO_2, has been noted. This might indicate that dissolution mechanisms are occurring at a higher rate than precipitation (Peng et al. (2022)). On the other hand, quartz is not a mineral of quick reaction (Wigand et al. (2008), and Othman et al. (2018)), which indicates that in a 30-day period, no changes should be noted. In this context, the increment produced in quartz content could be related to the reduction in content of other minerals, which would proportionally increase its content in the big picture.

When referring to the variation produced in the plagioclase, the same changes were observed by Cui et al. (2017) and Peng et al. (2022), the first researcher indicating a reduction in the anorthite and an increment in the albite, while the second author presented a general decrease in plagioclase content. As it might be observed from Figure 3, the plagioclase present in the Banco Verde sandstone is characterized for being composed of anorthite in greater proportions. This is the case presented by Hangx et al. (2009), in which it is demonstrated that anorthite reacts with water saturated CO_2, producing calcite and clay minerals consequently. Even when the XRD do not show an increment in calcite content, the Bernard Calcimetry test was performed in pristine and carbonated specimens, and an increase in the carbonate content was observed.

3.3. Discussion

To begin with, it is important to note that, as it was expected, the degree of variation of the properties of the sandstone were more notable in the pieces of sandstone obtained from the edge of the core, rather than the ones obtained from the centre. This is mainly due to the difference in the exposure time to CO_2. It should be highlighted that even when the centre was less altered than edge, CO_2 reached this zone during the aging period, as the sandstone's properties in this area were also altered.

To continue, according to Laskowski et al. (2023), from the analysis of X-ray fluorescence, it was concluded that clay mineral dissolution occurred when injecting CO_2. It was considered that the clays dissolved came from the glauconite, but having observed the thin sections, it would also be reasonable to affirm, that the clay grains present in the calcite cement in some grain contacts. This would contribute to explaining the reduction in the mechanical strength of the rock (Laskowski et al. (2023)), considering there is a modification not only in the grain-to-grain interaction but also in the rock matrix.

Finally, in terms of porosity and pore size distribution, some observations should be made. On the one hand, a reduction in the porosity was observed, being this previously related to fines migration and further precipitation in bigger pores, produced due to clay dissolution (Laskowski et al. 2023). This precipitation would produce a reduction in the bigger pores' diameter, increasing the number of smaller pores, and reducing the amount of ink-bottle pores, which could be observed in the micro structural analysis of the MIP tests. The reduction in porosity had also been related to the increment produced in carbonate content, which was shown to be produced due to the interaction between the plagioclase, more specifically the anorthite, and the water saturated CO_2.

4. CONCLUSION

CO_2 underground storage has proven to be a feasible alternative to mitigate climate change. In Argentina, the interest in this particular solution has increased over the past few years. In this context, the Banco Verde layer from the Salamanca Formation has been proposed as a possible storage for CO_2. With the aim of contributing to a better understanding of the interactions produced between CO_2 and the reservoir sandstone chosen, a series of micro structural and compositional analysis were conducted. Among the main conclusions reached, a reduction in porosity should be mentioned, as well as deposition of fines and carbonates, due to the reaction of CO_2 with clay minerals, glauconite and the anorthite in the plagioclase. This research would be enriched if it was complemented with a petrographic analysis of thin sections of 30-day carbonated sandstone and scanning electron microscopy (SEM) imaging, which would allow a more graphical understanding of the chemical and micro structural phenomena involved in the injection of CO_2 into the glauconitic sandstone studied.

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