

# **AN EXPERIMENTAL INVESTIGATION OF THE ROCK PROPERTIES EVOLUTION ASSOCIATED TO DIFFERENT LEVELS OF CO<sub>2</sub> INJECTION LIKE ALTERATION PROCESSES**

P. Egermann, E. Bemer and B. Zinszner  
Institut Français du Pétrole (IFP)

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## **ABSTRACT**

Ensuring well injectivity and integrity over the lifetime of CO<sub>2</sub> geological storage operations is crucial for both the technical and economical success of such projects. Another key point is the monitoring issue, which requires reliable parameters to convert seismic data into CO<sub>2</sub> saturation distribution maps. A specific difference between CO<sub>2</sub> injection and conventional hydrocarbon gas injection is the possibility of geochemical reactions between the mobile reactive brine and the host formation during CO<sub>2</sub> injection leading to modifications of the rock flow, geomechanical and petro-acoustical properties.

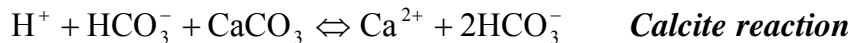
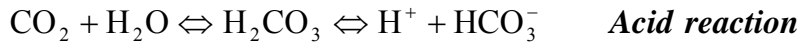
The main objective of the paper is to experimentally investigate the variation of these properties for several levels of rock alteration. We first present an innovative experimental procedure leading to a homogeneous dissolution pattern of the rock samples, avoiding especially any wormholing process that would drive to erroneous measurements at the core scale. The proposed approach relies on the injection of a retarded acid solution, which is activated only when in place under specific temperature conditions. Porosity, permeability, geomechanical and petro-acoustical measurements were performed both on a low and a high permeability set of carbonate samples, either under their native state or different states of alteration.

The results show that permeability evolution follows an increasing trend due to the dissolution process. Nevertheless, permeability increases are rather limited even in the case of very pronounced porosity increases (4-5 porosity units). This effect is attributed to the mobilization of fines that are destabilized when the rock alteration is severe. From the geomechanical and petro-acoustical points of view, altered samples usually show a decrease in stiffness and shear strength. To our knowledge, similar set of petrophysical measurements conducted on samples with different levels of rock alteration has not been published earlier. Therefore, this work will contribute to the development of the constitutive laws to be implemented in the geomechanical reactive transport simulators used to model the geological CO<sub>2</sub> storage.

## INTRODUCTION

### Rock/fluid interactions during CO<sub>2</sub> injection

One very specific feature of the CO<sub>2</sub> injection is the possibility for rock/fluid interactions. When injecting in the reservoir, which can be an aquifer or a waterflooded oil reservoir, one fraction of the CO<sub>2</sub> dissolves into the brine and forms H<sub>2</sub>CO<sub>3</sub>, which dissociates to give a weak acid that has the potential to react with the host rock formation (calcite, dolomite, anhydrite, etc ...).



### Evolution of the rock petrophysical, mechanical and petroacoustical properties

Taking into account the change in rock properties induced by a pore structure modification during a CO<sub>2</sub> injection is a key issue to capture the dynamic behavior of the reservoir:

- Petrophysical properties can significantly impact the injectivity and thus the pressure field in the reservoir,
- Mechanical properties control the stress sensitivity of the reservoir. Rock weakening induced by rock/fluid interactions can be detrimental to the well or the reservoir integrity (fracture activation, completion failure, ...),
- Relevant petro-acoustical properties are needed to interpret seismic data and monitor accurately the CO<sub>2</sub> distribution in the reservoir.

The permeability evolution induced by the flow of a reactive fluid has been extensively studied in the past to better model the well stimulation operations based on acidification (Bazin, 2001) and more recently in the context of the geological CO<sub>2</sub> storage. Rock/fluid interaction phenomena in presence of CO<sub>2</sub> have been observed both in laboratory and field studies on samples submitted to CO<sub>2</sub> injections. Reactive-transport experiments in presence of CO<sub>2</sub> have been performed both in sandstones (Ross et al., 1982) and carbonates (Svec and Grigg, 2001; Grigg and Svec, 2003). The results demonstrated that a modification of the rock structure can either improve or impair the permeability value depending on the driving geochemical processes (precipitation/dissolution reactions), which are functions of the rock fabric, the brine composition and also the thermodynamic conditions. Recent experimental investigations have evidenced that the flow regime also has a significant impact on the modification of the permeability since it controls the residence time of the reacting fluids within the porous medium (Egermann et al., 2005a). In the near wellbore region, wormhole dissolution patterns are observed due to the non equilibrium of the geochemical reactions. In the far field region, a homogeneous modification of the pore structure is expected because the dissolution regime is uniform.

These evolutions of the rock structure have also been observed from field tests. The thin section analysis of samples cored in the far-field region of a CO<sub>2</sub>-flooded area showed

dissolution of anhydrite (Mathis et al., 1984). Kane (1979) reported nearly 50% injectivity improvement attributed to the rock dissolution on the SACROC field. In other reservoir studies, Kamath et al. (1998), Christman and Gorell (1990) and Prieditis et al. (1992) did not record any rock/fluid interaction impact on the petrophysical properties demonstrating that this process is very case dependant.

The change in mechanical properties of a rock submitted to a mechanical damage induced by an excessive loading has been widely studied (Charlez, 1991), but the effects of a chemical damage due to rock/fluid interactions remain to be fully characterized. The problem can be assessed through a phenomenological approach by introducing an irreversible porosity induced by the chemical alteration and adopting expressions of the poroelastic parameters, which are functions of this irreversible porosity (Coussy, 2004). One of the objectives of the present work is to provide primary trends for the evolution of the elastic moduli and the uniaxial compressive strength with the increase in porosity induced by a chemical alteration.

To be relevant, the petrophysical, mechanical and petro-acoustical measurements must be performed on samples that can be considered as representative elementary volume to insure that the macroscopic petrophysical parameters are uniform at the core scale. This assumption is no longer valid for samples that are affected by wormhole dissolution patterns, which is the most common case in the laboratory studies due to the operating conditions (Svec and Grigg, 2001; Grigg and Svec, 2003; Egermann et al., 2005a; Izgec et al.). It is then essential to develop an adapted procedure to prepare altered samples in a controlled manner and with a homogeneous modification of the pore structure so that the macroscopic petrophysical and mechanical measurements provide a sound basis for modeling purposes.

## **Outline**

This paper presents an innovative methodology to alter rock samples in a controlled and homogeneous manner in order to investigate the evolution of the petrophysical, mechanical and petro-acoustical properties. The first part of the paper describes the experimental procedure leading to a homogeneous dissolution pattern of the rock samples, avoiding especially any wormholing process that would drive to erroneous measurements at the core scale. The proposed approach relies on the injection of a retarded acid solution, which is activated only when in place under specific temperature conditions. The second part is dedicated to the results of petrophysical, mechanical and petro-acoustical measurements performed both on a low and a high permeability set of carbonate samples, either under their native state or different states of alteration. Evolution of the different properties is discussed in each subsection. Finally, conclusions are drawn on the future works to be conducted.

## EXPERIMENTAL ALTERATION OF ROCKS

### Principle of the homogeneous alteration process

The understanding and the modeling of the wormholing process have been studied a lot to control and optimize the acidizing stimulation treatment of production wells (Daccord et al., 1989; Bazin, 2001). One of the major results of these studies is that the dissolution regime can be mapped as a function of two dimensionless numbers, the Peclet and Damköhler, in a  $P_e$  versus  $P_e D_a$  plot (Schechter and Gidley, 1968; Daccord et al. 1993; Bekri et al. 1995 and 1997):

$$P_e = \frac{v_0 l}{D}, \quad D_a = \frac{\kappa}{v_0} \quad \text{and} \quad P_e D_a = \frac{\kappa l}{D}$$

where  $v_0$  is the fluid velocity,  $l$  a characteristic length of the pore,  $D$  the molecular diffusion coefficient and  $\kappa$  the chemical reaction rate (Figure 1). In most laboratory cases, the regime is governed by wormholes or compact dissolution (sharp front of dissolution at the inlet face).

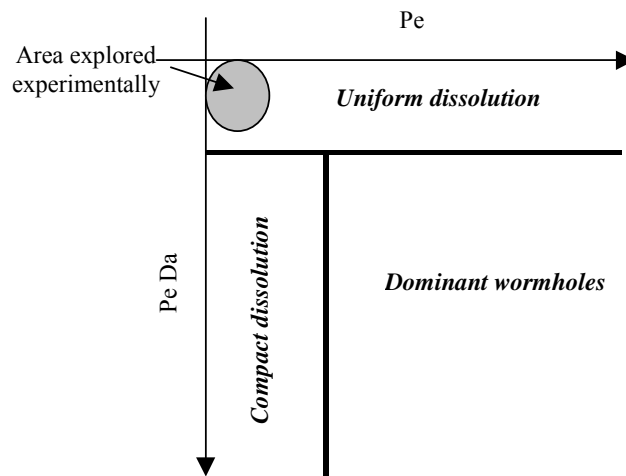


Figure 1: Dissolution regime as a function of the Peclet and the Damköhler numbers

The upper part of the graph is very interesting since it corresponds to a uniform dissolution regime of the pore space. In a previous paper, we presented an experimental procedure to alter homogeneously rock samples, which was based on the quasi-instantaneous placement of a reactive solution in the pore space so that  $P_e$  is roughly equal to zero (Egermann et al., 2005b). In this paper, we present an innovative approach for altering even long samples that relies on the injection of a retarded acid solution, which is activated only when in place under specific temperature conditions to avoid wormholing.

In-situ organic acid production systems are based on the use of "acid precursors" (water-soluble organic compounds that may be hydrolyzed to produce acid). Acid is produced at a predictable rate for a given set of conditions (i.e. acid precursor concentration and type, temperature, pH, presence of catalyst, concentration of catalyst). For carbonate dissolution, precursors that produce formic or acetic acid are generally the most useful. In-situ acid generating systems are widely used in oil industry applications where good zonal coverage is important, for example the treatment of filter cake in long horizontal openhole wells. They may also be used in other formation acidizing applications, such as matrix acidizing and permeability enhancement of natural fracture networks (Almond et al., 1995; Harris et al., 1998, 2001; Leschi et al., 2006)

## Data acquisition

### *Alteration experimental set-up*

It is mainly composed of a core holder cell, where one rock sample is mounted. The typical size of the sample is 40 mm in diameter and 80 mm in length to perform representative geomechanical experiments after alteration (length/diameter ratio higher or equal to two). The end-pieces of the core holder are connected to different equipments:

- A buffer cell used to place the retarded acid in the sample very quickly. The cell is filled initially both with retarded acid and pressurized nitrogen to conduct the injection at high, constant differential pressure,
- A brine injection system and a differential pressure transducer in order to remove the products of the reaction and also to measure the permeability values of the altered samples at different stages,
- A cleaning line connected to the inlet end-piece of the core holder to remove the retarded acid after its placement into the sample and avoid acid injection during the next injection through the sample,
- A fluid collector at the outlet to check the volume of retarded acid injected into the sample (roughly three pore volumes per treatment).

The principle of the apparatus is described in Figure 2. The differential transducer used to measure the evolution of the permeability as a function of the rock alteration degree is chosen according to the permeability value in order to work in the optimal range of the device. In the case of extremely high permeability, the differential pressure measurements were double-checked using one U-tube system based on gravity head differences. A Back Pressure Valve (BPV) was also mounted in all the experiments and was used during the cleaning periods, when fresh brine is injected to remove the products of the reaction. Because the acid reaction produces some CO<sub>2</sub> as a gas phase, the BPV enables to expel it very quickly from the sample by dissolution into the fresh brine. Therefore, only 5 pore volumes were needed to reestablish a fully saturated state with the initial brine for permeability measurement.

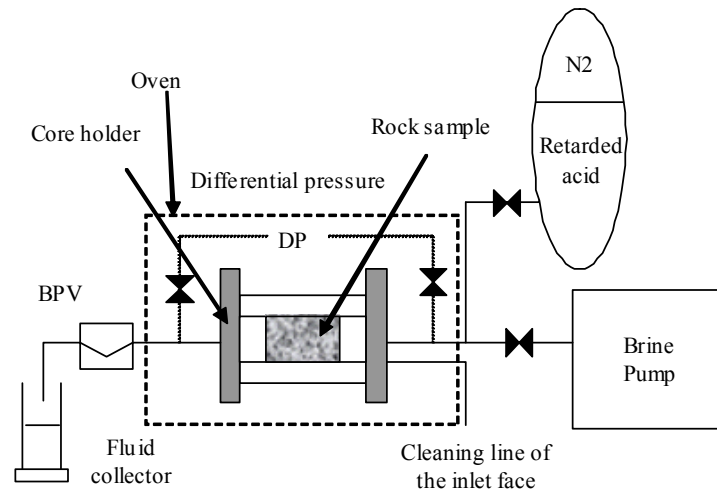


Figure 2: experimental apparatus used for homogeneous alteration

### Experimental procedures for alteration and subsequent petrophysical measurements

Specific procedures were developed to obtain both a good characterization of the sample prior and post the rock alteration and also a recording of the evolution of the permeability during the intermediate states of the rock alteration:

- Mercury porosimetry on companion plug,
- Dry weight,
- Saturated weight and evaluation of the initial porosity  $\phi^0$ ,
- NMR measurement for porosity check,
- Initial permeability measurement:  $k_w^0$ ,
- Retarded acid placement (3 PV),
- Cleaning of the inlet end-piece of the core holder with fresh brine to prevent injection of the activated acid located upstream the sample,
- Activation of the acid under moderated temperature (60-70°) for two days,
- New retarded acid placement or flush with fresh brine,
- Measurement of the permeability  $k_w^i$  corresponding to the  $i^{\text{th}}$  retarded acid treatment,
- Dismantle the sample when the expected permeability variation is reached,
- Saturated weight,
- NMR measurement for porosity check,
- Oven at 80°C for two days and dry weight,
- Mercury porosimetry of the altered sample.

Additional acid treatment

One advantage of this protocol is that the permeability variation can be followed continuously. Because the sample is never dismantled during the subsequent treatments to avoid breaking and failures (especially with fragile rock-types), only the overall porosity variation can be measured using this protocol, it can be assumed that the

intermediate variations of the porosity is directly proportional to the treatment number since the same volume of acid is placed during each treatment.

## EVOLUTION OF THE ROCK PROPERTIES

### *Petrophysical properties*

The alteration quality has been evaluated through CT-scanner measurements of the porosity profiles prior and post the chemical treatment. Figure 3a demonstrates that the alteration procedure based on the quasi-instantaneous acid placement (Egermann et al., 2005b), which was successfully applied on slices of rock samples (length in the order of 2 cm), is no more applicable with long samples (8 cm). Altered porosity profile exhibits strong end effects with no porosity evolution in the center of the sample. This effect results from the surface to volume ratio, which is less favorable for long samples compared to sliced-shape samples.

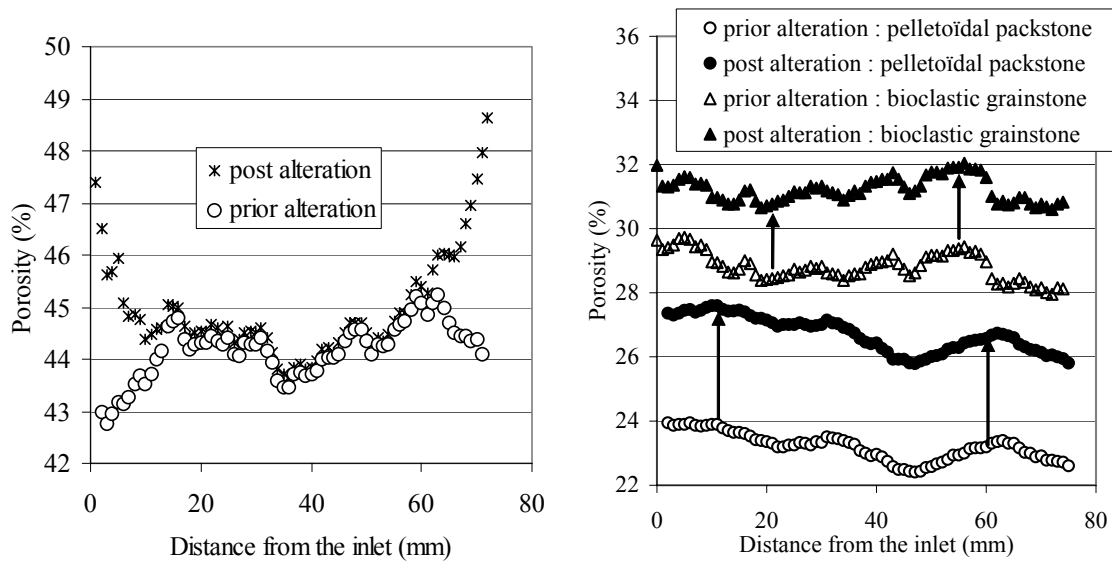


Figure 3 : (a) Porosity profiles using a quasi-instantaneous acid placement on long cores – (b) Porosity profiles using the retarded acid on long cores (two rock-types)

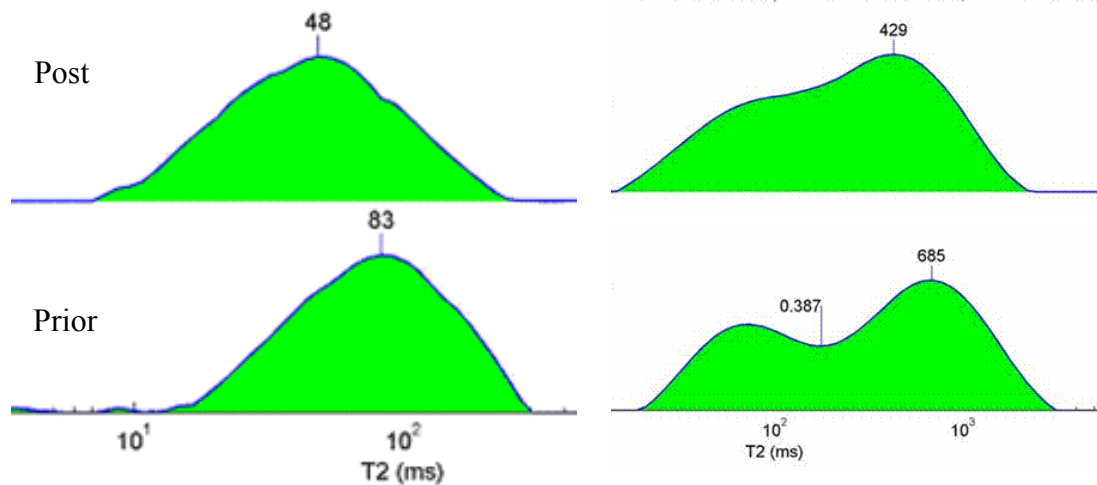
The experimental protocol based on retarded acid injection gave systematically homogeneously altered porosity profiles as shown in Figure 3b for two carbonate rock-types (pelletoidal packstone and bioclastic grainstone). Depending on the number of repeated treatments (roughly 0.4 porosity increase is obtained by treatment), the overall porosity increase can be as many as 4 porosity units (Table 1). Although significant porosity evolutions are recorded, the associated permeability increases are limited (between 3 and 15%). This result can be related to experimental observations made during the preparation of the samples. When displaced from the core to the separator, the acid solution that reacted with the rock was found systematically very dense and charged with fine particles in suspension. This observation is certainly associated with the

mobilization of fines destabilized by the small scale rock alteration that can impair the permeability in the downstream part of the core.

*Table 1 : Evolution of the average porosity and permeability (initial and final stages)*

Sample	$\phi^0$ (%)	$k_w^0$ (mD)	$\phi^{\text{final}}$ (%)	$k_w^{\text{final}}$ (mD)
Pelletoïdal packstone	24.3	2.92	27.8	3.03
Bioclastic grainstone	28.8	146	31	171

Evolution of NMR T2 distributions were observed for both rock-types with a shift to lower values meaning that the ratio V/S decreased (Volume/Surface). Because the porosity increase leads to higher volume values, these results are certainly related with an increase of the specific surface due to the small scale dissolution process. This trend is also in line with results obtained on altered samples under reservoir conditions with CO<sub>2</sub> (Egermann et al., 2005a).



*Figure 4 : Evolution of the NMR T2 distribution with the alteration process (left : pelletoïdal packstone, right : bioclastic grainstone)*

### **Geomechanical properties**

At this stage of the study, geomechanical properties were simply determined through uniaxial compression tests. These tests are easy to conduct, but the results are dependant on any default and/or heterogeneity of the sample. In the second stage of the study, geomechanical properties of both intact and altered samples will be measured in a triaxial cell under various confining pressure, which will ensure relevant measurements and give access to the whole shear strength criterion.

The samples were altered in the petrophysical experimental set-up and dried after cleaning. They were then equipped with six strain gauges: three for the axial strain measurement and three for the radial strain measurement. The key parameters chosen to follow the effect of the chemical alteration are the uniaxial compressive strength ( $UCS$ ), the tangent drained bulk modulus ( $K_o$ ) and the tangent shear modulus ( $G$ ).



A first set of intact pelletal packstone samples has been tested to evaluate the intrinsic scattering of the rock geomechanical parameters. Samples showing several alteration levels have then been studied. Figure 5 shows the evolution of the selected geomechanical parameters plotted against the sample final porosity (initial porosity plus additional irreversible porosity induced by the chemical alteration). Figure 6 reports the same evolution plotted against the irreversible porosity increase.

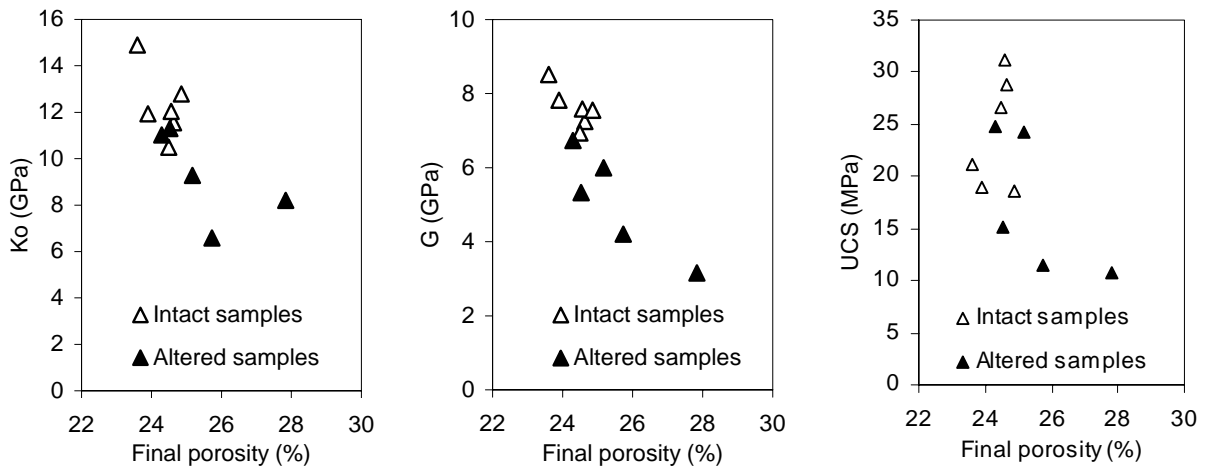


Figure 5 : Geomechanical properties of the altered samples plotted against the final porosity

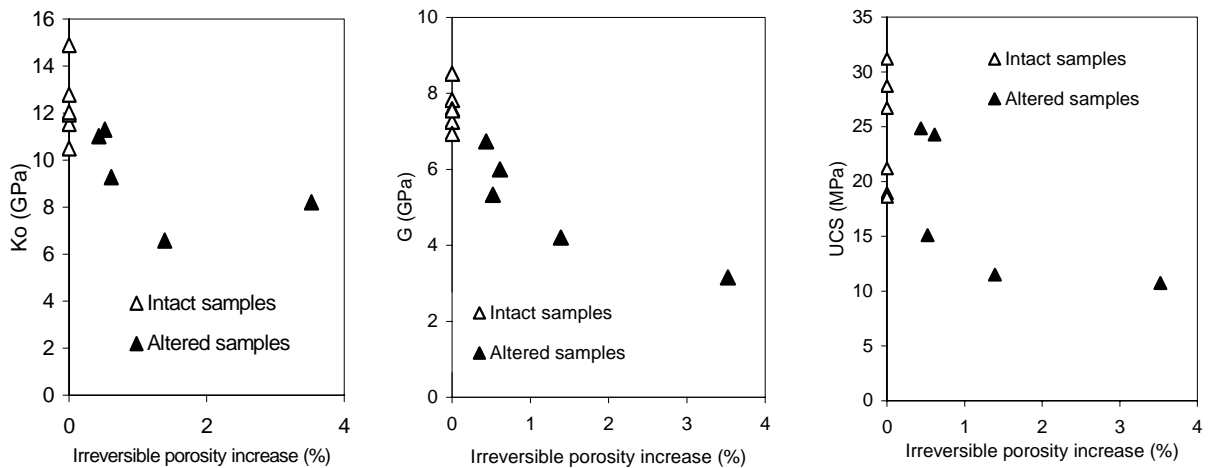


Figure 6 : Geomechanical properties of the altered samples plotted against the irreversible porosity increase

As expected, the rock strength and stiffness show a decrease with the porosity increase. The weakening is more pronounced for the shear modulus than the drained bulk modulus, which suggests a significant alteration of the cementation in accordance with the UCS reduction. The key point is then to determine if the properties of the altered samples are of the same magnitude as the properties of equivalent intact samples with the same porosity.

Bemer and al. (2004) have proposed a phenomenological model leading to expressions of the drained bulk modulus and the shear modulus as functions of the porosity and calcite intrinsic moduli. Empirical correlations between the shear strength parameters and the porosity have also been given. Figure 7 shows how the altered samples fit in the general trends. In spite of the natural scattering of the experimental data, we observed a more pronounced weakening with the porosity increase for the samples chemically altered.

These first results need to be extended, but we can already note that models based on a global porosity value will a priori fail to assess limestone chemical alteration and its effect on the cementation.

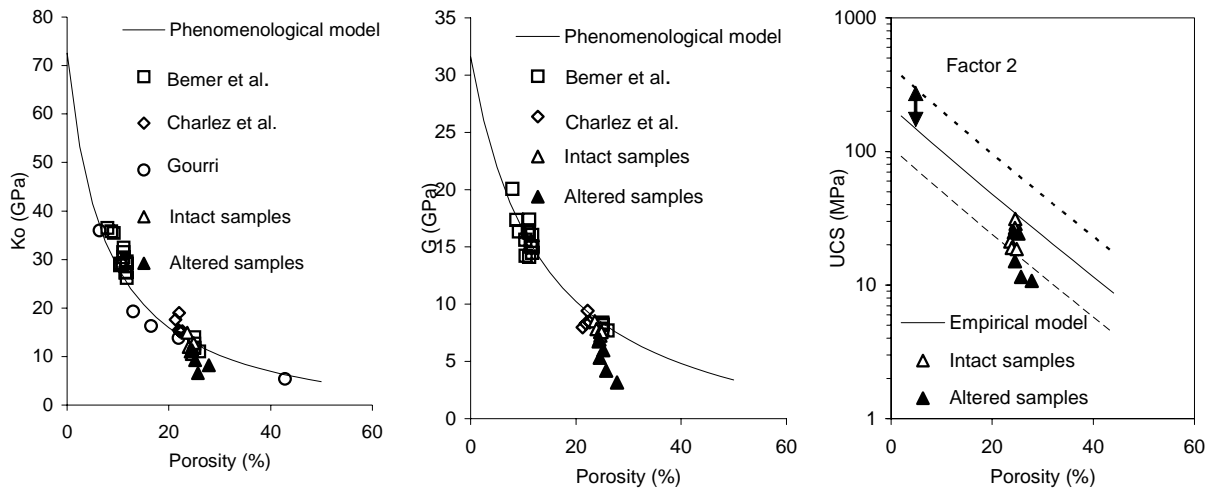


Figure 7 : General trends for the evolution of the geomechanical parameters of intact limestones as a function of their porosity

### Petro-acoustical properties

The experimental procedure for acoustical properties acquisition is basically the one described above for dissolution process but, in order to precisely measure the small variation in ultrasonic wave velocity at each dissolution step, the acid generation is performed inside the acoustic cell (Figure 8). The acoustic measurement device consists in two Compressional (P) and Shear (S) piezoelectric transducers located inside steel boxes (measuring head) to protect them from confining pressure. The rock sample is maintained between the measuring heads by a Viton® sleeve. Both measuring head have a hydraulic connection to outside the cell to perform liquid substitution inside the sample. Ultrasonic compressional (VP) and shear (VS) velocities have been measured, using both first break picking and the phase delay (Bourbié et al. 1987). The values presented in Figure 9 correspond to the later technique, which is more reliable for limestone (Rasolofosaon and Zinszner, 2004).

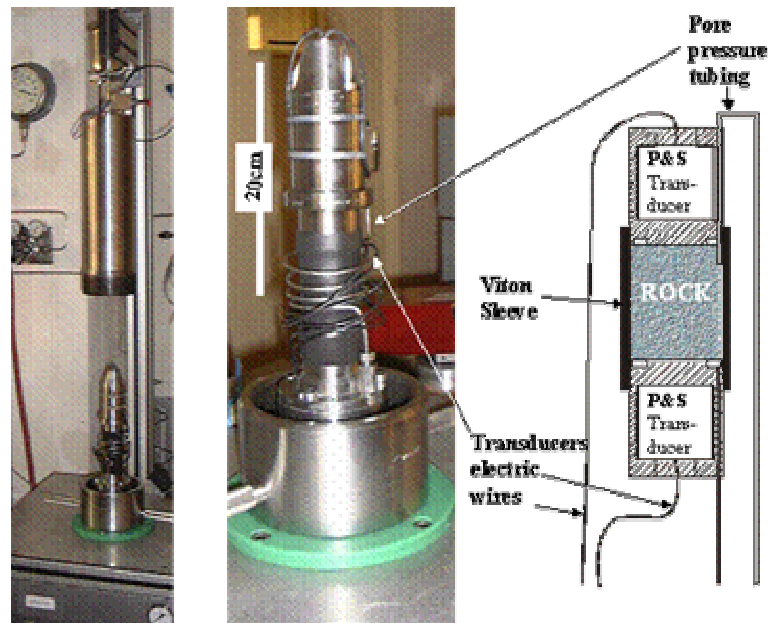


Figure 8 : Pressure cell for petroacoustic measurements (photographs and sketch)

When dealing with the impact of dissolution on wave velocity, the important point is not the absolute value of velocity variation but the relative velocity decrease related to porosity increase. Indeed, the relationship between velocity and porosity has been extensively studied (see Mavko et al., 1998, for a review) and we know the usual "transforms" between these variables. Two situations have to be considered: either the velocity variation observed during progressive dissolution is consistent with the porosity increase and we can conclude that no specific phenomenon impacted the rock microstructure during dissolution, or the velocity variation is much greater than expected. The occurrence of the latter case has to be carefully taken in account for an interpretation of acoustical data.

Both situations were observed and are summarized in Figure 9 where the solid lines correspond to the slope of the empirical "Field Observation Transform" statistically derived for limestone (Schlumberger co, 1989). For legibility of the figure, the absolute values of the transform are translated.

The slope of the velocity variation in the pelletal packstone sample is as expected from the usual transform. But the slope for the bioclastic grainstone sample is much greater. Although the CT-scanner observation confirmed the homogeneity of the dissolution at the millimeter scale in both samples, most probably at the infra millimeter scale in the bioclastic grainstone, the dissolution impacted selectively the intergranular porous system while the intragranular microporous system was less concerned due to a less efficient liquid displacement inside the micropores.

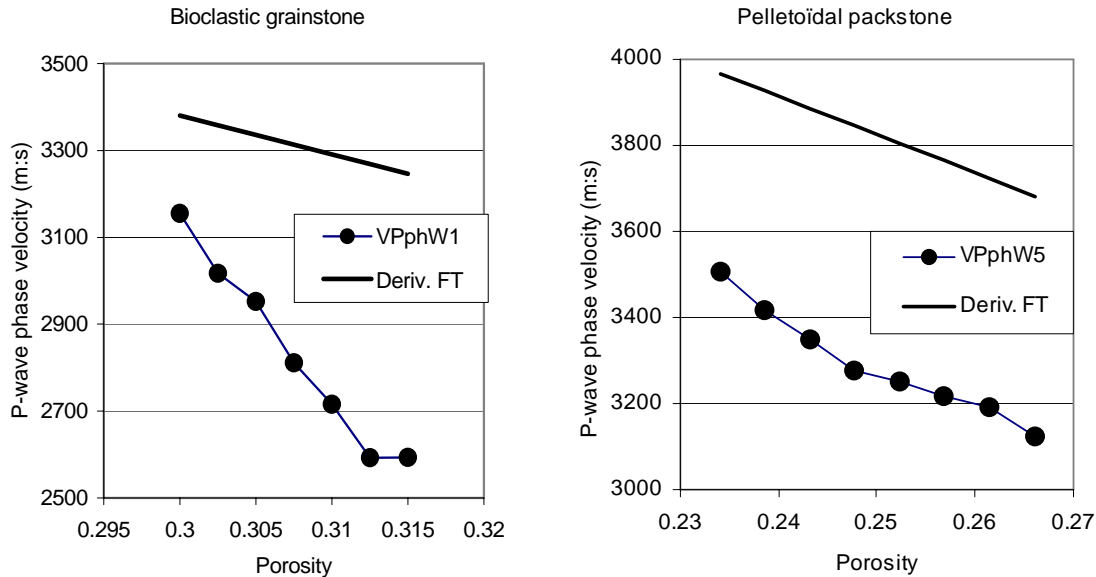


Figure 9 : Variation of P-wave velocity with porosity increase. The solid line correspond to the slope of the empirical "Field Observation Transform" for limestone (translated values)

Concerning acoustical properties, the relative impact on velocity of dissolution induced porosity increase exhibits contrasting situations. When the dissolution is supposed to be homogeneous including the microporous system, the velocity variation is "normal" i.e. consistent with the usual empirical transforms. But in other case, the impact could be much greater than expected when related to porosity variation. This situation could be greatly misleading when performing interpretation of acoustical data and deserves a careful investigation. An inhomogeneous dissolution at the infra millimeter scale could provide a first explanation.

## DISCUSSION AND CONCLUSION

An innovative approach is proposed to alter homogeneously the pore structure of carbonate rocks. The method can be applied on various sample sizes and was used to investigate the impact of the CO<sub>2</sub> like rock/fluid interactions processes on petrophysical, mechanical and petro-acoustical properties. The experimental results evidence that the evolution of these properties can vary significantly toward what is predicted by phenomenological models based on the evolution of the "natural" porosity:

- permeability evolution can be impacted by the mobilization of fines destabilized by the small scale rock alteration. Fines mobilization can therefore reduce the expected increase of permeability for a given value of porosity increase,

- mechanical properties evolutions show a more pronounced weakening with the porosity increase for the samples chemically altered because of its effect on the rock cementation. This behaviour is also in line with the mobilization of fines process,
- petro-acoustical properties evolutions were found contrasting according to the initial rock structure. Whereas pelletal packstone behaviour follows a "normal" trend, the bioclastic grainstone behaviour deviates significantly, meaning that misleading interpretation of seismic data could be done if the effect of the pore structure alteration is not taken into account.

To our knowledge, similar set of petrophysical measurements conducted on samples with different levels of rock alteration has not been published earlier. Therefore, this work is a contribution to the development of the constitutive laws to implement in the geomechanical reactive transport simulators used to model the geological CO<sub>2</sub> storage. Further tests will be conducted on other rock-types to identify the most sensitive initial pore structures to rock/fluid interactions.

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