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VACA MUERTA PORE SYSTEM. WHAT WE´VE LEARNED?

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RESUMEN

Sistema Poral de Vaca Muerta. MEB, SGA, RMN T1-T2
El objetivo de esta contribución es compartir nuestro entendimiento actual del sistema poral de la formación Vaca Muerta, a partir de la integración de numerosos estudios de laboratorio sobre roca y perfiles especiales. Es un complejo sistema de poros típico de las formaciones de lutitas ricas en materia orgánica, que combina los poros de la matriz inorgánica y los poros alojados en la materia orgánica sólida. El soterramiento y los procesos diagenéticos de la formación controlan la variación de la forma, el tamaño y la conectividad de cada tipo de poro. El sistema poral resultante está directamente relacionado con la capacidad de almacenamiento y producción del fluido.
En esta contribución, comenzaremos por una breve revisión de la clasificación general de poros en lutitas orgánicas y luego nos enfocaremos en la formación Vaca Muerta. Varias de las técnicas de laboratorio disponibles, y sus limitaciones asociadas, serán brevemente mencionadas y se ilustrarán con ejemplos de Vaca Muerta. A continuación, mostraremos cómo las mediciones de perfiles eléctricos especiales pueden ofrecer información sobre este sistema de poros, lo que permite una interpretación continua a lo largo del pozo.

INTRODUCTION

The types of pores and their distribution and connectivity are major controlling factors for storage, permeability, and wettability (Loucks, *et al.* 2012). But, how different can pores be? By pores, we mean a space in the formation that is not matrix. In organic-rich formations, pores can be hosted within both inorganic (IMphi) (clay, silicate or carbonate minerals) and organic matrix (OMphi). Many variables impact pores: stratigraphic position and their associated different depositional energy surely could imply pore changes. Besides the original deposition setting, diagenesis will preserve or transform the primary porosity, compaction, and can reduce size of pores; Thermal maturation and hydrocarbon generation will create pores in the organic matter; their size and connectivity being driven by maturity level and organic chemistry, if different macerals are present. Elias and Gelin (2015) identified different organofacies in the large vertical extent of Vaca Muerta.

Pore size and pore throat dimensions must be compared to the size of the fluid molecules. Size matters! Oil molecules are bigger than gas molecules. can both oil and gas molecules move through the pore system? Defining where the bigger and more connected pores are, is particularly relevant to understanding the hydrocarbon production.

Intra-porosity is generally less connected than inter-porosity and organic pores. Within organic matter hosted pores, different connectivity can be found as well. In addition, superficial effects in organic-hosted pores can be detrimental to production. As Jarvie (2015) mentions, inorganic pores yield better gas or oil retrieval because sorption is reduced in the absence of original organic matter. In an organic-rich shale, sorption results in retention of petroleum.

Many studies have demonstrated that Vaca Muerta pore system is complex and varies through the well and with basin position (Reed, *et al.* 2017; Crousse *et al.* 2015).

High TOC is not always synonymous with productivity, especially when the TOC is mainly associated with organic matter (OM). This has led to the definition of alternative practical reservoir quality indices, for example the Reservoir Producibility Index (RPI) (Kausik *et al.* 2015).

For these reasons, it is essential when evaluating an organic-rich formation to reach the best possible control and understanding of the pore distribution within the formation, and their relation to permeability for different phases.

Methodology

There are several techniques to analyze pore size, type and shape. Each one has its own limitations and ranges of measurement. In this study several techniques available today are utilized, keeping in mind that there is no perfect methodology, laboratory, sample preparation, scale and quantification method. Each technique provides a partial characterization of the rock, and by integrating, complementing and contrasting different set of data a more robust interpretation can be achieved.

In this study a complete set of laboratory analysis (XRD, XRF, Leco, Pyrolisis, Porosity, Subcritical Nitrogen Gas Absorption, 2D & 3D Scanning Electron Microscope) was done in 76 rotated sidewall cores from two vertical wells which cover the range from peak oil to condensate window. Wells have a full logging suite in the Vaca Muerta interval, including spectral gamma ray, nuclear magnetic resonance, elemental spectroscopy & dielectric dispersion. The objective of the study is to characterize the Vaca Muerta pore system integrating all existing data, but also to understand how far the interpretations of pore types and distribution can be pushed based on each source of information to later try and apply the acquired knowledge in areas with sparser available information.

In the following sections, a brief description of each source of information is described, focusing on what information they provide, their limitations and how it can be used.

ROCK LABORATORY STUDIES

Scanning Electron Microscope (SEM)

Scanning Electron Microscope (SEM): SEM data is critical to understand the pore system of an organic-rich rock; it's the only technique that enables a direct visualization of where the porosity is hosted (pores in the organic matter or the inorganic matrix), its type (intergranular, intercrystalline, intragranular, etc.), size and shape. The downside of this method is the extremely narrow area covered by a single SEM image. To gain representativeness, many samples for a given interval or facies must be analyzed.

Another consideration for interpretation is that SEM measurement of pores can be sub estimated since some heavier hydrocarbons could be in solid state at surface conditions. However, downhole these heavier hydrocarbons are in a fluid state, being part or the pores, not of the matrix.

The SEM images for the current project were prepared using the ion beam milling method, which emphasizes material density, allowing an easy identification of pores, organic material and mineral grains (Figure 1).

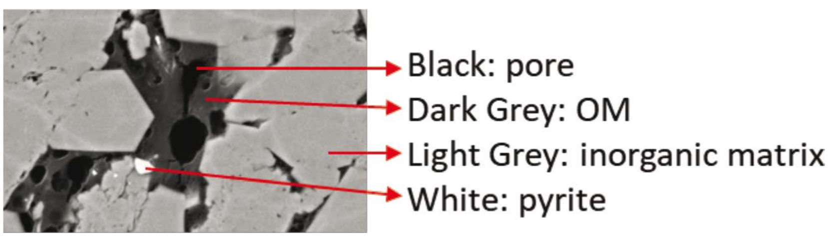

Figure 1. SEM image

The SEM images in this study cover an area of 254um x 219um, with a pixel resolution of 4.1nm distinguishing pores bigger than 15nm. Estimation of organic matter, total porosity, organic porosity, mineral porosity, pore shapes, and pore sizes were obtained after image analysis. Organic and mineral pores were separated by pixel classification, allowing a quantification of each pore type volume (Figure 2).

Image analysis were conducted using proprietary software. All area fraction was computed based on the total area (2 dimensional of the non-mineral material). From SEM images, the empty pores are shown as nearly black, whereas the mineral matrix is represented by different shades of gray, indicating differences in material density that are a result of grain density. The dark-gray material is commonly interpreted as organic matter.

Under reservoir conditions, kerogen is a solid particle of unconverted organic matter, whereas bitumen and oil are liquids. When the viscosity of bitumen and oil is high, they display the same gray-scale intensity as solid kerogen under laboratory SEM observations (Zhang *et al.* 2017)

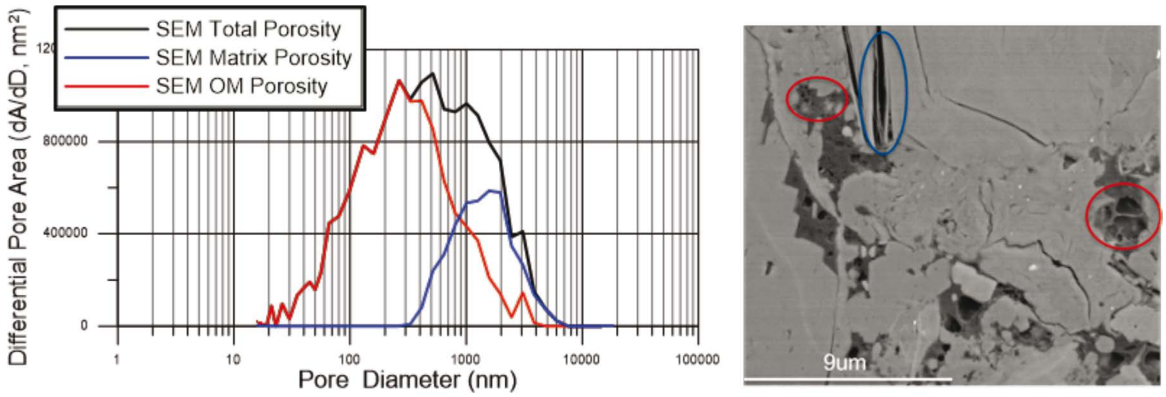

Figure 2. On the left, Pore Size Distribution (PSD) from a sample where the black curve represents total pores, blue curve inorganic pores and red curve organic pores. On the right, SEM image illustrating organic pores (circled in red) and inorganic pores (circled in blue).

A subset of samples was further used for 3D SEM (Figure 3). Despite the sparseness of the information, the resulting general tendencies of organic matter amount and organic vs inorganic pore type predominance were aligned with the ones observed by other techniques (2D SEM, LECO and NMR T1-T2). As a result, the information provided by 3D SEM was not used quantitively but integrated as support for other observations. In addition, the 3D SEM still helped to separate different vertical sections in Vaca Muerta using the relative fraction of connected pores.

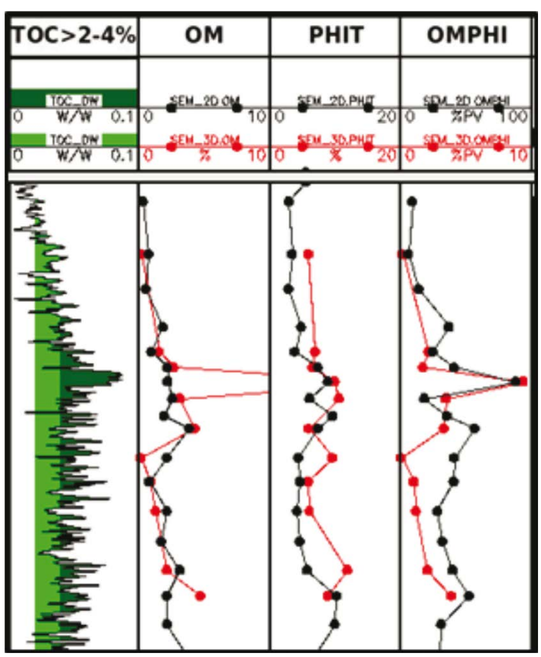

Figure 3. Red dots represent 3D SEM samples, and black dots 2D SEM samples. Track 1: TOC (w%). Track 2: organic matter from SEM quantification. Track 3: Total porosity from SEM. Track 4: Organic porosity (OMphi) from SEM.

Subcritical Nitrogen Gas Absorption (SGA)

This study provides a pore size distribution for each sample and the evidence of the presence or not of small organic pores (range between 2-40nm approximately). Interpretations on SGA give an idea of where pores are mainly hosted (inorganic matrix or organic matter).

SGA is a laboratory-based technique where nitrogen at 77.3K is intruded into the sample and the pressure and gas quantity injected are recorded as isotherms (Fischer *et al.* 2018; Kuila *et al.* 2014; Saidian *et al.* 2014; Kuila *et al.* 2012). These isotherms are the base for calculating the connected pore size distribution. SGA can measure pores as small as 2nm, turning it in a powerful study for shale analysis where there is a significant amount of very small pores. Unfortunately, the technique is not capable of measuring pores bigger than 200nm resulting in a limited range of measurement.

The SGA study can be complemented by repeating the measurement in the sample after the organic matter has been removed by NaOCl solvent (Kuila *et al.* 2014). By comparing results before and after organic matter removal, valuable information on organic matter porosity (OMphi) presence and amount can be obtained. For details of methodology refer to Fischer *et al.* (2018).

Three SGA groups (Kuila *et al.* 2014) can be observed in Vaca Muerta samples:

Group 2, where pore size distribution (PSD) curves as received and after organic matter removal are parallel and distant to each other. This implies that after organic matter (OM) removal, porosity increased almost throughout the entire pore size range (red star in the figure 4). Group 2 matches with TOC values less than 2wt% and is interpreted as a rock with no development of organic pores, due to lack of organic matter or not enough thermal maturity.

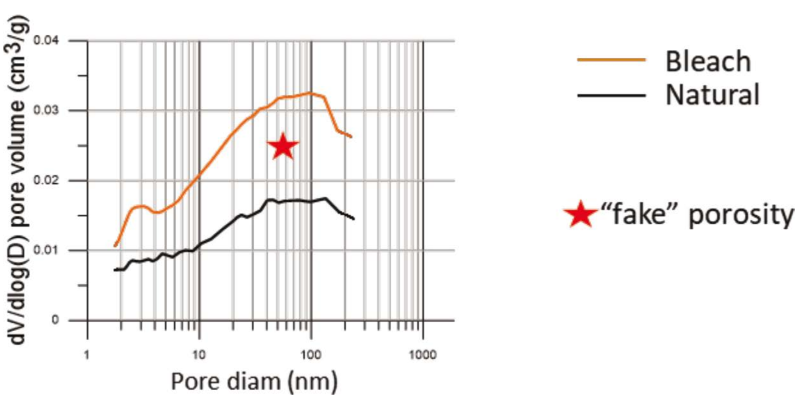

Figure 4. PSD example for Group 2.

Group 2-3, where pore size distribution (PSD) curves as received and after organic matter removal present a small crossover around 3nm. The subtle reduction of small porosity could be related to some organic pores (green star in figure 5). These samples present TOC values between

2 to 4 wt%. Group 2-3 is interpreted as a rock with little to no development of organic pores smaller than 200nm, due to not enough thermal maturity. This rock could present pores bigger than 200nm but fall out of methodology limits.

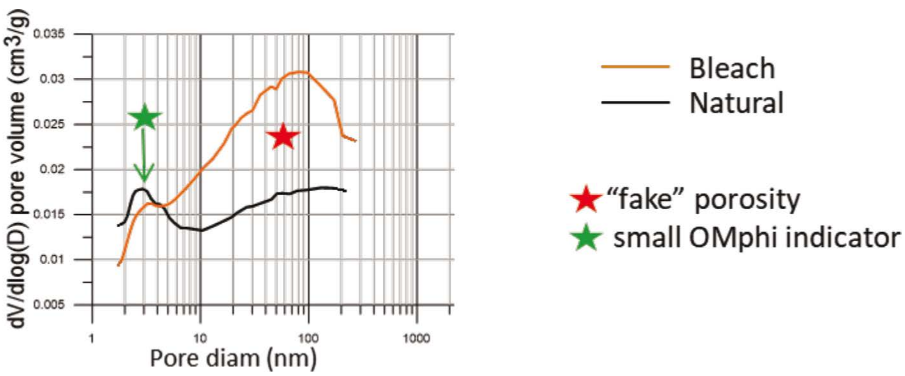

Figure 5. PSD example for Group 2-3.

Group 3, where PSD curves as received and after organic matter removal present a significant crossover. The reduction of small porosity suggests that those pores (green star in figure 6) were hosted in the organic matter. These samples present TOC values higher than 3 wt%. Group 3 is interpreted as a rock with a significant development of small organic pores. It is important to stress that bigger organic pores than 200nm could be there too.

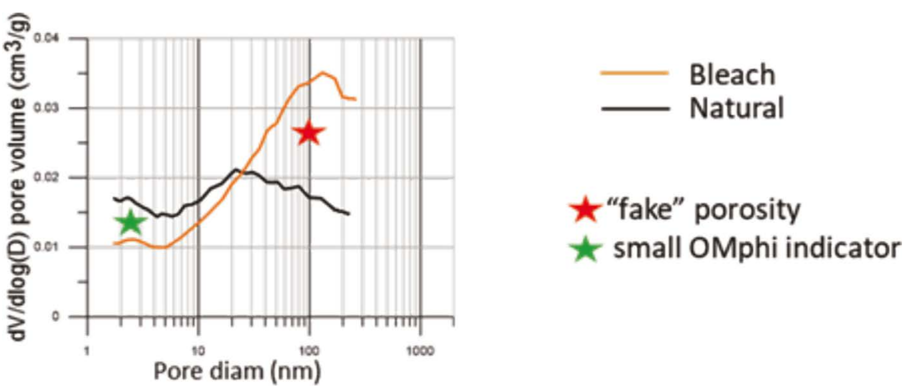

Figure 6. PSD example for Group 3.

Another piece of information provided by SGA study is the Specific Surface Area measured (SSA) by BET Theory (Brunauer *et al.* 1938). The specific surface area is the area of solid surface per unit mass. In practice, only the accessible surfaces are measured. In unconventional rocks, high surface areas tend to represent samples with either high amounts of clay or organic matter (OM). Typically, large amounts of OM will obscure the contributions to SSA from clay. SSA from OM tends to increase with increasing thermal maturity, as more and smaller pores are created.

E-LOGS

Nuclear Magnetic Resonance (NMR)

NMR is the measurement of choice to investigate the pore system. The distribution of transverse relaxation times T2 is driven by the pore surface-to-volume ratio, and by the fluid type filling and wetting the pore surface. Examples of use of NMR T2 distribution in Vaca Muerta can be found in reference Crousse *et al.* (2015). By defining a moveable fluid T2 cutoff, it is possible to estimate a free fluid index, a volume fraction of fluids that lies in the larger pores, that is expected to be related to early production.

Separating the pore and fluid types based on single dimension T2 distribution is not easy. Indeed, the contributions of different types of pores and different fluids may, and indeed do, overlap (Nicot *et al.* 2015; Sun *et al.* 2016). Complementing the T2 measurement with either

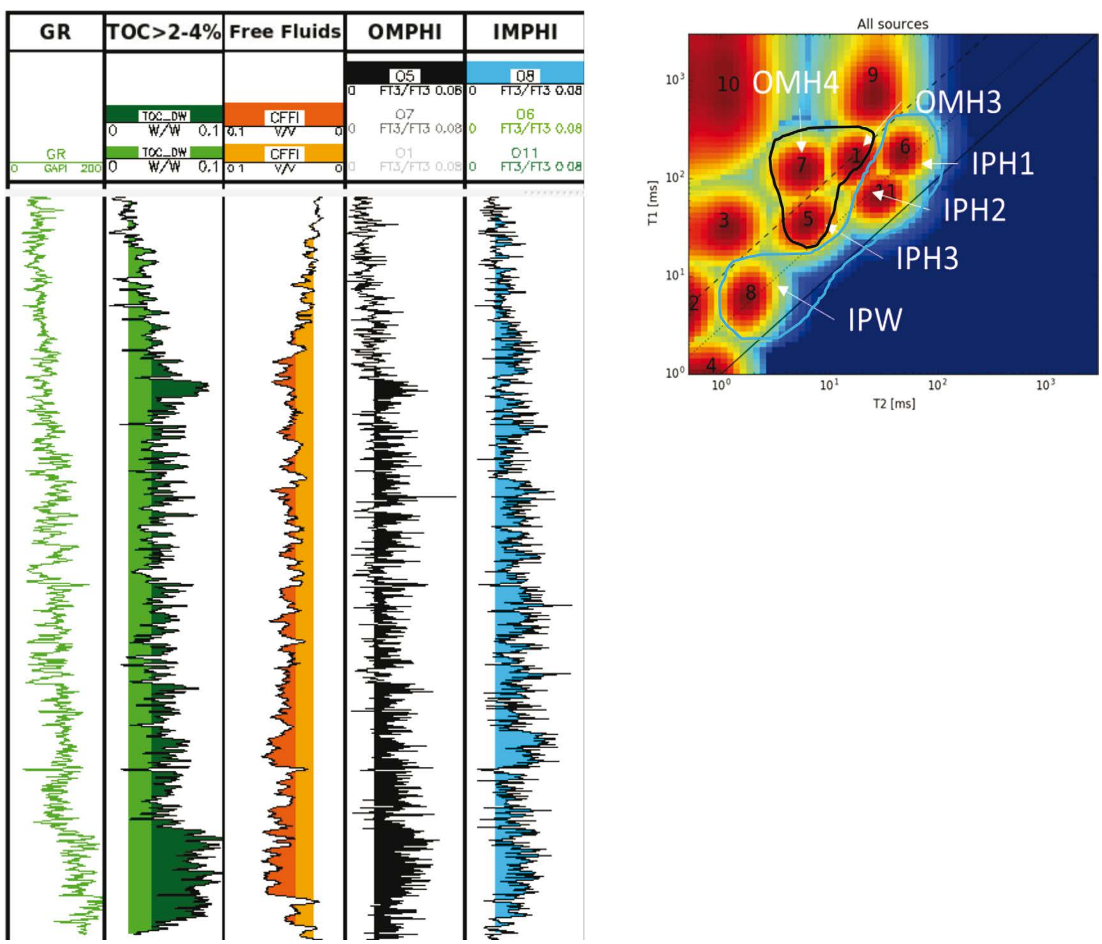

Figure 7. On the left, Vaca Muerta well were Track 1 shows: gamma ray; Track 2: TOC (w%); Track 3: Free Fluids from NMR; Track 4: Organic Porosity interpreted from NMR T1-T2 map and Track 5: Inorganic porosity interpreted from NMR T1-T2 map. On the right, T1-T2 map for the entire organic interval (Modified from Ortiz *et al.* 2017). Light blue line groups inorganic pores filled with light hydrocarbons and IP water. Black line, organic pores, filled with light hydrocarbons.

or both diffusion (Cao Minh *et al.* 2012) and longitudinal relaxation T1 (Jiang *et al.* 2013) helps deconvolute the situation. The recent developments in 2D T1-T2 measurements and analysis indicate that this latter method is the most efficient to separate fluid type and pore type (Ortiz *et al.* 2017). In Vaca Muerta, the pore water is usually found at low T2 and low T1/T2 ratios, while hydrocarbon hosted by the inorganic porosity shows at largest T2, and hydrocarbon hosted in organic porosity is usually found at low T2 but higher T1/T2 (Figure 7).

The vertical resolution in typical NMR tools is at best about 1 foot, and commonly about 2-3 feet. This vertical resolution must be accounted for when comparing to core measurements.

Dielectric Dispersion

Dielectric dispersion logs measure the variations of the complex permittivity (relative permittivity and conductivity) over microwave frequency range, typically in the MHz to GHz range. Both relative permittivity and conductivity are driven by the amount of water in the formation, the salinity of this water, the geometrical shape of this water and to the clay superficial effects (especially at low water salinity). One of the output of the dielectric dispersion analysis is a textural parameter representing the geometry of the water in the formation (parameter called MN in this paper). This textural parameter is then driven by the shape of the pores hosting the water.

The type of matrix hosting the pore water, either carbonate, siliciclastic or clay may impact MN. The presence and thermal maturity of organic matter is also expected to affect MN. Finally, the presence of conductive minerals, such as pyrite, or conductive by-product of organic matter maturation, such as graphite, may also influence MN (Misra *et al.* 2016.). A detailed investigation on the main factors driving MN in organic-rich formation is still pending. Observations from a growing number of wells indicates trends of MN with maturity that is still to be deciphered, not only for the piece of information it conveys about the pore system, but also because it impacts the conventional resistivity interpretation (Ortiz *et al.* 2018).

RESULTS

In Figure 8 different laboratory studies and e-logs are shown for a Vaca Muerta well. Each one provides partial and complementary information.

Vaca Muerta pore system varies significantly. From SEM images it can be observed that interparticle pores are around mineral and/or kerogen particles. Porosity associated to clay flakes and between coccolith debris is common. Intraparticle pores can be found in less stable crystals and in pyrite framboids. Organic pores can be observed in a variety of shapes (spherical and elongated) and sizes. In intervals where TOC is higher than 5w%, the organic matter is distributed throughout the matrix. While in sections with less TOC the OM is generally present in patches or strings.

Inorganic vs. Organic porosity predominance can be studied using quantification from 2D SEM. That information can be used to validate the NMR T1-T2 interpretation, enabling to extrapolate pore type estimation to non-sampled intervals.

By integrating SGA and SEM, measured pore size range goes from 2 nm (SGA technical limit) to more than 1000nm. Most of the pore area comprises pores around 130 and 800nm. IMphi is bigger than OMphi. OMPHI average diameter is smaller (40-200nm) than IMphi (200-1500nm). OMphi generally increases size with depth, due to maturity and larger OM patches. Bigger IMphi is associated with clays (within clay platelets) and intercrystalline pores. In the lower section, OMphi ranges from 40-80%. In the upper section, IMphi generally predominates over OMphi (OMphi: 20-40%). IMphi seems to be more developed in the siliciclastic matrix than in the carbonatic one. Higher BET values indicate predominance of smaller pores.

SGA provides information about the generation or not of small organic pores; suggesting that those pores are developed depending not only on thermal maturity but also on organic matter content.

Another piece of information is the texture of the pores filled with water, coming from the MN from Dielectric Dispersion log. This data is still under analysis, but so far, higher values of MN

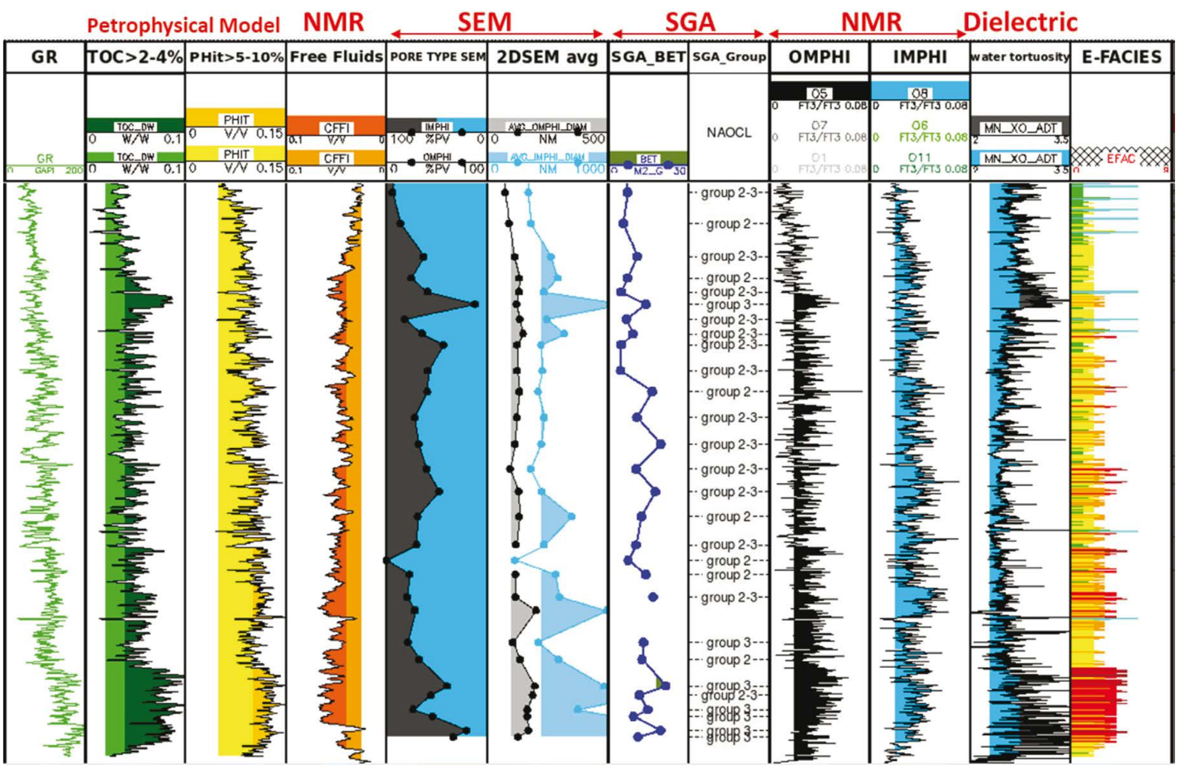

Figure 8. Vaca Muerta well showing vertical variation of reservoir properties analyzed by different studies. Track 1: gamma ray; Track 2: TOC from petrophysical model. TOC higher than 2w% in light green and higher than 4w% in dark green. Track 3: Total porosity from petrophysical model. PHIT higher than 5w% in yellow, higher than 10w% in orange. Track 4: Free Fluids from NMR, darker orange represents bigger volume. Track 5: Pore type % from 2D SEM, OMphi (dark grey) and IMphi (light blue). Track 6: OMphi and IMphi average pore diameter, increasing towards the right. Track 7: BET from SGA, increasing towards the right. Track 8: SGA group classification. Track 9: OMphi pore volume from NMR T1-T2, Track 10: IMphi pore volume from NMR T1-T2. Track 11: MN from Dielectric Dispersion log, higher values towards the right. Track 12: Electrofacies.

match with higher OMphi. This provides independent information that confirms observations made on SEM, SGA and NMR.

After obtaining a good characterization of the pores using lab results and logs, an upscale to electrofacies was made to predict the pore system in other wells. As explained in detail in Crousse *et al.* (2018, this Conference), the 8 electrofacies used in this study were generated for the Vaca Muerta-Quintuco interval and represent different reservoir properties such as TOC, porosity and carbonate content. Figure 9 shows 7 electrofacies (1 electrofacies has no rock sample) and their distinct pore systems.

Organic matter (OM) varies in abundance and aspect, from being present throughout the matrix in warmer color efacies, to being represented as scarce grains in colder color efacies. Pore type relative predominance also, changes significantly. Inorganic pores (IMphi) clearly dominates in the colder efacies while organic pores (OMphi) are very significant in orange, red and brown efacies. Regarding dominant pore size range, in colder efacies both, OMphi and IMphi are small to medium. Yellow efacies starts showing big IMphi pores, while dominant OMphi are still medium size. In orange, red and brown efacies, small OMphi mode appears, coexisting with medium to big OMphi size; while IMphi size can be small and big.

EFacies	OM	Pore Type	OMPHI Pore Size	IMPHI Pore Size
Brown	Lot through the matrix	50% OMPHI 50% IMPHI	Lot small (2-40nm) & Big (>300nm)	Lot small inter/intraparticle Big (>600nm) between clays
Red	Lot through the matrix	50% OMPHI 50% IMPHI	Lot small (2-20nm) & Big (>300nm)	Lot small-med inter/intraparticle Lot Big (>600nm) between clays
Orange	Patches	40% OMPHI 60% IMPHI Connected pores	Lot small (2-20nm) & Lot med (>250nm)	Lot small-big-very big interparticle Lot Big (>600nm) between clays
Yellow	Few in patches	30% OMPHI 70% IMPHI Connected pores	Lot med (>250nm) No small range (2-40nm)	Lot small & Few med-big interparticle Med-Big (>600nm) between clays
Green	Scarce	20% OMPHI 80% IMPHI Connected & non connected pores	Med (80-200nm)	Small & med interparticle
Cyan *Blue*	No to Scarce	10% OMPHI 90% IMPHI Non connected pores	Med (100-200nm)	Small interparticle

Figure 9. Table summarizing main characteristics of pore system for each electrofacies defined in Vaca Muerta-Quintuco interval for the studied area.

CONCLUSIONS

Vaca Muerta in the studied area contains a mix of mineral and organic matter-hosted pores. Vertical variation in pore type, quantity, size and connectivity suggest different "play types" within Vaca Muerta organic section. This leads to the existence of numerous possible landing points.

Mineral-hosted pores dominate in different intervals and organic matter-hosted pores become increasingly important in deeper sections and where TOC is higher.

In general, organic pore size increases downwards, while inorganic pores size is more variable. Over imposed on the general trend of increasing size of organic pores with thermal maturity, the appearance or not of a small organic pore type is observed, which seems to be more associated to a different kerogen type or a difference in kerogen amount which leads somehow to the generation of those kind of pores. The two sections in the well where those pores are observed correspond to transgressive deposits. These could lead one to think there is a relation among the depositional environment, the anoxic conditions, the amount of the organic matter, and the amorphous type preservation.

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