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## Use of advanced NMR measurements to estimate key subsurface properties of organic rich shales: the case of the Vaca Muerta formation

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### Abstract

The development of organic rich shale formations poses a set of challenges, originating from the lack of a strong correlation between traditionally accepted subsurface static parameters, such as total porosity or STOIP, and well performance. Complex porosity systems, consisting not only of pore space formed during deposition but also of pores formed as a result of different diagenetic processes, fluid composition defined not only by the balance of capillarity and buoyancy but also by volumes of hydrocarbons generated *in situ* during organic matter maturation, and high formation ductility, causing very rapid permeability decline during production, require a different set of formation static properties to estimate the relative efficiency of hydrocarbon recovery for these formations. Such properties need to be quantified reliably in formation core samples and from downhole measurements.

In this paper, it is demonstrated how advanced NMR measurements, in combination with other core analysis techniques, can be used to assess the static properties of organic-rich shale formations going beyond total porosity and water saturation in core samples and their further upscaling to the logs. Particularly, NMR measurements performed on as-received core samples combined with high quality volumetric measurements, allow us to quantify Bulk Volume Water (BVW) and volumes of residual oil occupying matrix and organic matter pores. NMR data acquired during pressure saturation experiments, combined with as-received NMR data, allow to estimate matrix porosity and organic matter porosity. The same experiment, combined with liquid permeability measurements, allows to quantify the volume of fluid occupying the microfracture network and the NMR signal corresponding to such fluid. The use of H<sub>2</sub>O-D<sub>2</sub>O exchange NMR experiments allows to quantify the bulk volume of bound water and formation water salinity. All these advanced NMR measurements, in combination with other core measurement techniques, allow to develop an NMR data interpretation scheme applicable to downhole measurements, such that the formation parameters mentioned above can be quantified at the reservoir scale.

The results of advanced NMR measurements, accompanied by volumetric and liquid permeability measurements performed on twin plugs for a large set of Vaca Muerta core samples, are presented. Such static parameters like BVW and Bulk Volume Hydrocarbons (BVHC) in matrix and organic matter pores and in microfractures are derived from these NMR measurements and are correlated to permeabilities of the microfracture network and formation porosity system. Also, the same static parameters were derived from NMR logs, and a systematic core–log comparison was performed. An approach for evaluating these static properties for wells where NMR logs are not available is discussed.

## Introduction

The Vaca Muerta formation is one of the most prolific unconventional plays in the world both in terms of thickness, areal extent, depth, organic content, thermal maturity level (González et al 2023) and in terms of observed performance of the development wells, which could demonstrate as high initial production rates as 5000 bbl/day. The very high concentration of present-day organic matter (up to 12 wt % (González et al 2023)) indicates an overcharged nature of this source rock, which differentiates it from organic matter lean formations of other liquid rich shale plays. Also, the porosity structure of the Vaca Muerta formation clearly demonstrates the presence of well-developed organic matter porosity together with different types of matrix pores and clay porosity (Crousse et al 2015). Such a complex porosity system poses a set of unique challenges for the petrophysical evaluation of this formation, which has to go beyond total porosity and water saturation in the total porosity space. Another challenge is related to the interconnectivity of different parts of the porosity system defining fluid flow in the formation and, as a result, the permeability model allowing to predict formation flow properties and how they may change with reservoir depletion. Eventually any petrophysical model developed using core data should be upscalable to the logs so that reservoir-scale sub-surface models could be developed and used to predict well performance.

It should be pointed out that the only formation characterization technique which can be used both in the lab and downhole and which has a direct sensitivity not only to the total volume of fluids present in the formation but to the volumes of fluids occupying different parts of the porosity system is Nuclear Magnetic Relaxometry (NMR) (Ellis 2007). Such sensitivity is provided by the transverse relaxation contrast for such fluids caused by the differences in pore surface – fluid molecule interaction. As it was shown before, NMR measurements allow to quantify BVW and BVHC in matrix porosity for lean Organic Rich Shales (ORS) (Nikitin et al 2017) and to exclude oil occupying organic matter pores, which is not producible due to the small size and the lack of interconnectivity of organic matter pores for that formation. Such separation of non-producible oil enabled the mapping of reservoir intervals with high hydrocarbon production potential.

As it was mentioned above, the Vaca Muerta formation contains a very high concentration of organic matter. Moreover, for a black oil maturity window portion of the basin organic matter contains significant fraction of extractable organic material generating continuously light hydrocarbons and as a result forming an active petroleum system. So because of the present day “production” of light hydrocarbons very high pore pressure should be expected (Cuervo et al 2018). Moreover, to allow new light hydrocarbon migration from the porosity system of the Vaca Muerta formation, *in situ* pore pressure should be expected to be very close to the lithostatic pressure. As a result, the presence of microfracture network filled with hydrocarbons should be anticipated. Such microfractures may provide a fluid flow path parallel to the fluid flow through the porosity system significantly impacting the behavior of the producing wells.

So, the evaluation of key petrophysical properties of the Vaca Muerta formation in the black oil maturity window has to be based on the use of advanced NMR measurements accompanied with other core analysis techniques so volumes of fluids occupying different parts of the porosity system could be quantified both in core samples and through downhole measurements. Also, the volume of water which

cannot flow (BVW\_bound) need to be estimated and such properties of formation water as its salinity need to be quantified. Finally, the flow properties of the reservoir including the role of microfractures in fluid transport through the formation need to be assessed.

## Theory and Methods

The Vaca Muerta formation contains a very high concentration of organic matter. As a result, the volume of generated HC is substantially higher in comparison to the water volume deposited, when the matrix porosity system was formed. Therefore, except the formation of well-developed Organic Matter (OM) porosity inside OM inclusions observed in SEM images (Crousse et al 2015) the network of OM coated channels should be formed providing the long-range interconnectivity of the OM porosity system. This part of the porosity system is coated by carbonaceous material and as a result it is ultra oil-wet. Also, it is filled with hydrocarbons *in situ* and does not contain any water. Such an assumption is valid because all hydrocarbons occupying the porosity system were generated as a result of the decomposition of a portion of OM during its maturation process. Because lighter HC have lower density HC generated in source rock occupy more volume when OM feedstock material and they are constantly ejected from OM pores. As a result, local pressure in OM pores is higher than the pressure in the matrix part of the porosity system and no fluid can be transferred from matrix pores to OM pores. Hence, both fluids located within the water-wet matrix porosity and ultra oil-wet OM porosity can migrate through the formation (see Figure 1) to fracture interfaces.

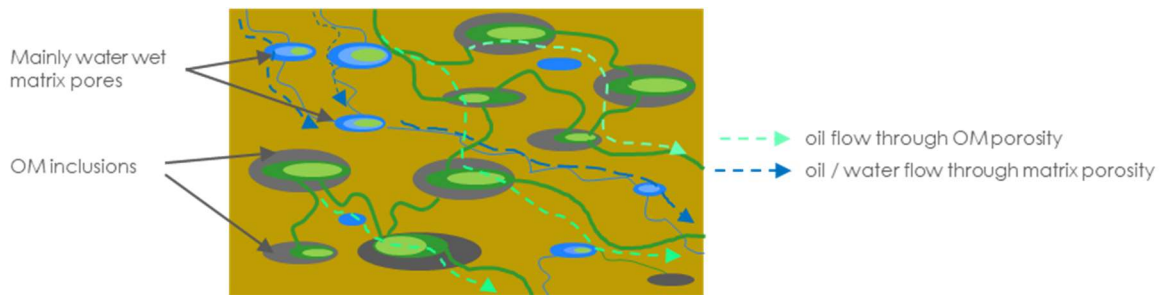


Figure 1: Conceptual model for the porosity system of Vaca Muerta formation.

The main mechanism of HC recovery in the Vaca Muerta formation is HC expansion caused by a decrease in bottom hole pressure due to fluid drainage. Because of its porosity structure and fluid composition described above, HC originally occupying OM pores can migrate to the hydraulic fracture interface either through the OM porosity network or through the matrix porosity network. For fluids occupying matrix porosity (water and light HC) the only path for the migration available is through the matrix porosity system itself (see Figure 2). It should be pointed out that if a microfracture network is present in the formation it will provide additional paths for fluid migration not shown in Figure 2.

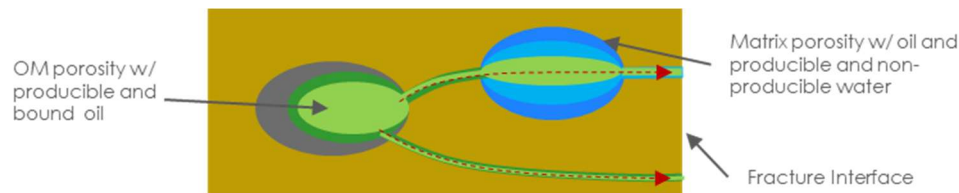


Figure 2: Conceptual scheme illustrating fluid migration through Vaca Muerta formation porosity system.

A volumetric component model for the Vaca Muerta formation can be described using the scheme illustrated in Figure 3. The model has three main parts:

- The organic matter (OM) inclusions, consisting of solid or immovable OM (immovable total organic content: TOC\_imm) and producible and non-producible HC occupying OM inclusion

pores (bulk volume hydrocarbon in organic matter:  $\Phi_{OM}$  equal to the sum of  $BVHC_{OM\_non\_prod} + BVHC_{OM\_prod}$ );

- The mineral matrix
- Matrix porosity ( $\Phi_{matrix}$ ), containing both bound and free water (bulk volume water:  $BVW$ ) and HC present in matrix pores (bulk volume hydrocarbon in matrix pores:  $BVHC_M$ ).

As it was pointed out above and discussed in Nikitin et al 2017 such separation of OM porosity and matrix porosity is required due to the different producibilities of fluids occupying these two components of the porosity system. As a result, the petrophysical evaluation model of such reservoir should account for  $\Phi_{OM}$  and  $\Phi_{matrix} / BVW_{prod} / BVHC_M$  separately.

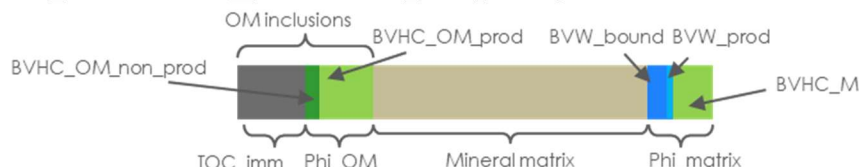


Figure 3: Volumetric component model of Vaca Muerta formation.

To collect core data which would enable the development of a quantitative petrophysical model for the Vaca Muerta formation (including the scheme allowing it to upscale to NMR logs) special core analysis workflow illustrated in Figure 4 was developed.

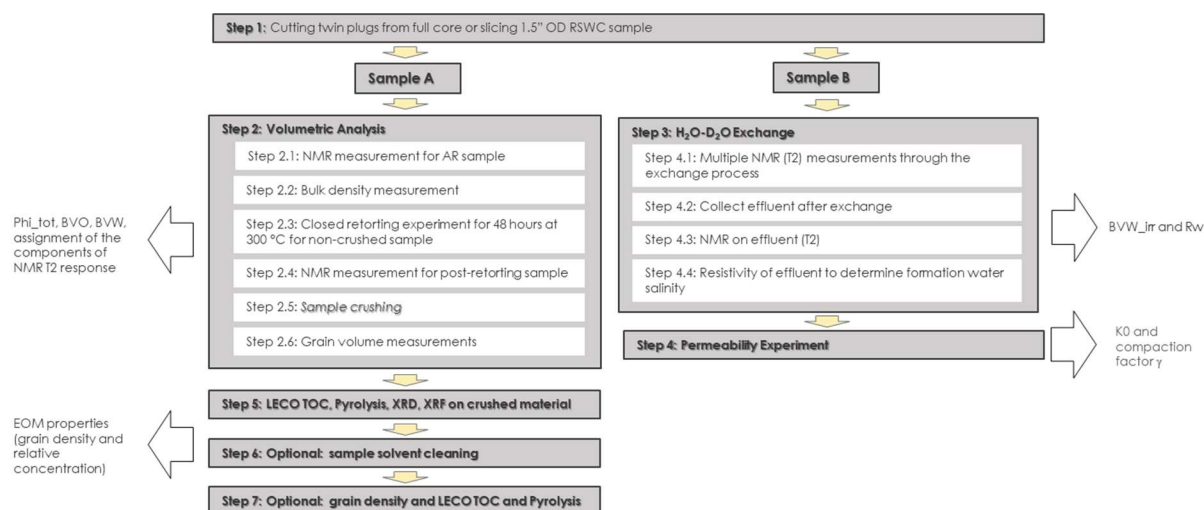


Figure 4 Scheme illustrating core analysis workflow developed to characterize Vaca Muerta formation core samples.

It requires twin samples either cut from the full core or sliced from a large Rotary Side Wall Core sample with a 1.5" OD. Sample A is used for volumetric analysis, which is destructive but does not change the fluid content of the sample, and Sample B is used for the H<sub>2</sub>O-D<sub>2</sub>O exchange experiment and permeability measurements, which modify the fluid content of the sample porosity system.

The volumetric analysis portion of the workflow uses a closed retorting experiment (Nikitin et al 2019), a fluid extraction technique, which does not require sample crushing, run at 300 °C for at least 48 hours. The sufficiency of the chosen duration of the retorting experiment was verified using periodical sample weight measurements for a small subset of samples. The fluid extraction based on the use of the whole sample implemented in this workflow allows to avoid fluid losses, caused by the crushing, which are very difficult to control and may depend on many factors including the lab environment and details of the sample crushing procedure. Examples of the impact of fluid losses caused by the crushing on volumes of extracted water are shown in Figure 5. These cross plots clearly illustrate that fluid losses caused by crushing introduce additional random and systematic errors, which are lab dependent and cannot be

corrected in a consistent way. Particularly crushing-induced fluid losses create a systematic underestimation of BVW resulting in a significant overestimation of BVHC and STOIP parameter of the unconventional reservoir under consideration.

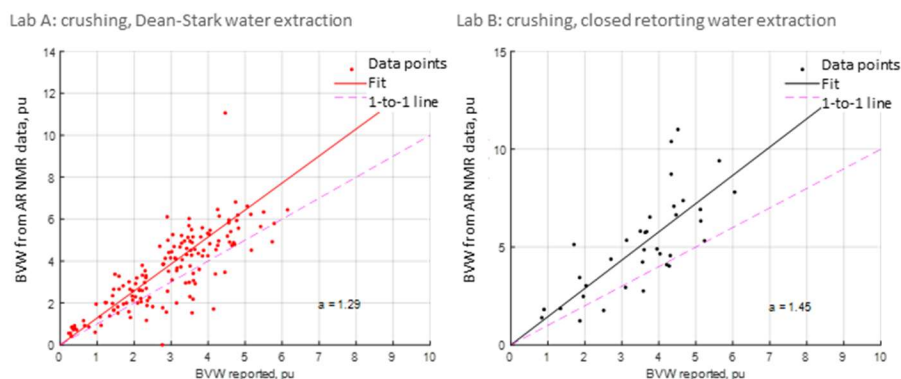


Figure 5: Comparison of volumes of extracted water performed for large sets of organic rich samples after crushing with BVW estimated using NMR measurements performed for as-received (AR) samples.

After NMR data for the post-retorting sample are acquired, samples are crushed, and sample grain density is measured using specially designed porosimeter which provides an accuracy of grain density measurement better than 0.0015 g/cc. Such high accuracy ensures that total porosity values calculated using data acquired by this workflow are accurate and do not suffer from systematic biases.

NMR data acquired for as-received and post-retorting samples combined with BVHC and BVW, measured using a closed retorting experiment, allow to assign components observed in the NMR response of core samples to fluids in different parts of the porosity system. Particularly, the differential NMR response of the core samples derived from the difference between an echotrain for the as-received sample and an echotrain for the post-retorting sample is only due to fluids that can be extracted through the retorting process at 300 °C. So, by correlating intensities of NMR components derived from differential echotrails, using Forward Modelling Based Inversion (Nikitin et al 2017), to BVHC and BVW values, the nature of such components can be identified. Figure 6 shows NMR T2 responses of the Vaca Muerta core samples (black oil maturity window) derived from echotrails acquired for as-received samples, from the echotrails acquired for post-retorting samples and the difference between these two echotrails, constituting a differential NMR signal. Here Non-Negative Least Square fit based inversion with Tikhonov regularization was used to convert different echotrails into T2 distributions. For all three sets each T2 distribution can be presented as a superposition of three components or peaks located in different T2 intervals. Correlation of peak intensities, derived from differential NMR T2 signal using FMBI, indicates that Peak 1 corresponds to water and Peak 2 and Peak 3 correspond to residual oil occupying different parts of the porosity system (see Figure 7). Because the average T2 value for Peak 3 is significantly higher than the average T2 value for Peak 2, Peak 3 should be assigned to oil in matrix porosity, which is at least partially water-wet, and Peak 2 should be assigned to oil in organic matter porosity, which is ultra oil-wet (Nikitin et al 2017). Also, a significant NMR intensity is observed in the Peak 1 T2 range for NMR T2 distributions derived from the echotrails acquired for post retorting samples. This peak correlates with TOC measured by the LECO method (see Figure 8), indicating that it is due to the hydrogen present in the solid OM. So, in the case of NMR data acquired for as-received samples Peak 1 is due to water and solid OM, Peak 2 is due to oil in OM pores and Peak 3 is due to oil in matrix pores. It should be pointed out that due to the very high pressure of the fluid in the pores of an organic rich shale reservoir, a significant portion of native fluid present *in situ* is lost by the core samples during their recovery (Nikitin et al 2017). Such loss is caused by HC expansion and as a result total porosity is systematically higher than volumes of fluids extracted from the core samples ( $\Phi_{tot} > BVW + BVHC$ ). Moreover, it should be assumed that the better interconnected part of the porosity system should lose more fluid occupying it *in situ* during the core recovery process.

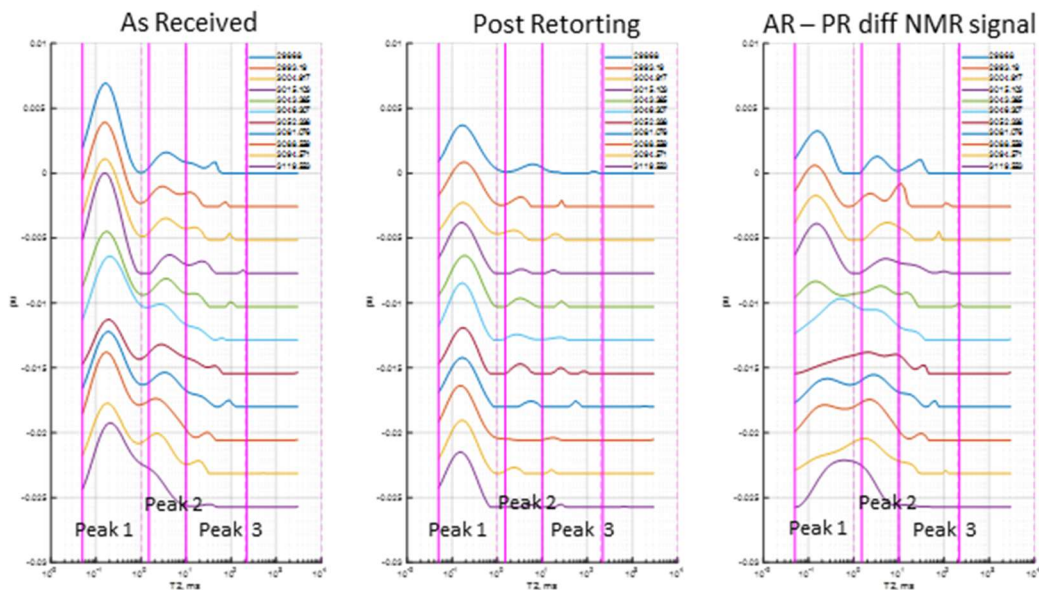


Figure 6: NMR T2 responses of Vaca Muerta core samples (black oil maturity window) derived from echotrans acquired for as-received samples, from the echotrans acquired for post-retorting samples and for the difference between these two echotrans (from left to right). Non-Negative Least Square fit based inversion with Tikhonov regularization was used.

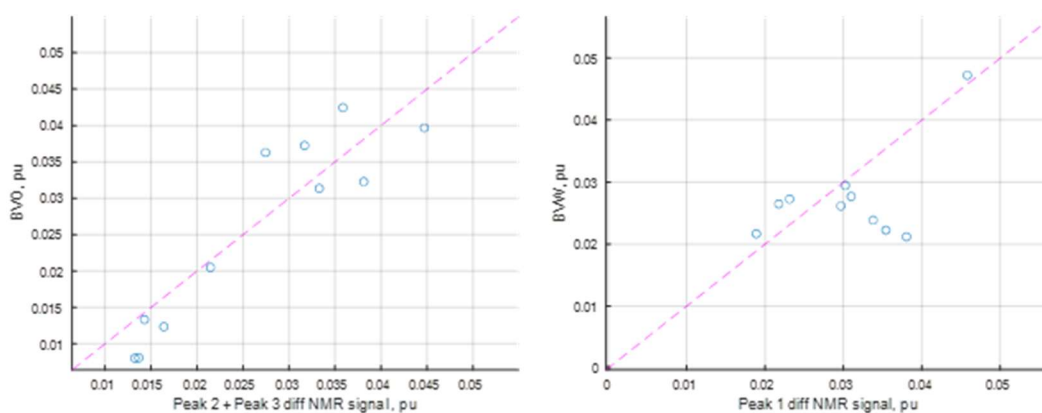


Figure 7: Comparison of the sum of Peak 2 and Peak 3 components derived from differential NMR T2 signal using FMBI with BVO from closed retorting experiment (left) and comparison of Peak 1 derived from differential NMR T2 signal using FMBI with BVW from closed retorting experiment (right).

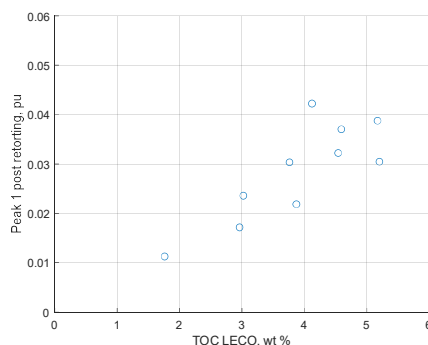


Figure 8 Intensity of Peak 1 for post retorting samples vs LECO TOC

Thermal extraction removes all fluid from the core samples and does not allow to separate water that can flow (BVW\_free) from the water that cannot flow (BVW\_bound). One of the methods allowing to assess the lower limit of BVW\_bound value (volume of water that cannot flow under any circumstances) is based on the isotopic exchange of native H<sub>2</sub>O molecules present in the core samples with D<sub>2</sub>O molecules, introduced from the outside. In comparison with hydrogen deuterium is NMR silent and as a result time-lapsed NMR measurements performed on the core samples exposed to D<sub>2</sub>O (heavy water) are going to be sensitive only to the evolution of H<sub>2</sub>O species occupying the sample porosity system. In this case H<sub>2</sub>O-D<sub>2</sub>O isotopic exchange drives H<sub>2</sub>O molecules, which are available for such exchange outside of the sample to the surrounding D<sub>2</sub>O liquid. An example of the evolution of the NMR signal during such an experiment for a Vaca Muerta core sample is shown in Figure 9.

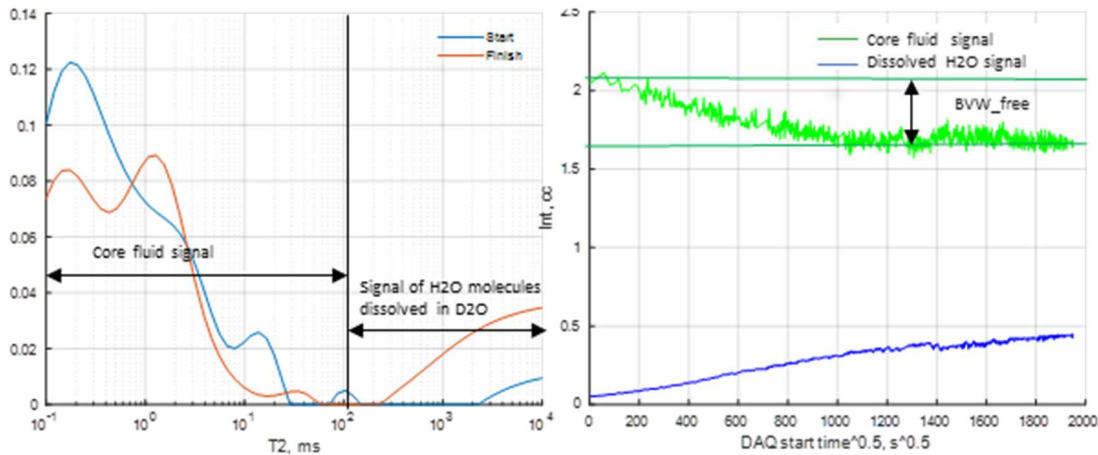


Figure 9: An example of the evolution of the NMR signal for a Vaca Muerta core sample exposed to D<sub>2</sub>O : NMR T<sub>2</sub> distributions in the beginning and at the end of H<sub>2</sub>O-D<sub>2</sub>O exchange experiment (left) and an evolution of the core fluid signal part and a portion of the signal which is due to H<sub>2</sub>O molecules dissolved in heavy water surrounding the core sample (right).

From this figure it can be seen that NMR T<sub>2</sub> distributions acquired in the beginning and at the end of the H<sub>2</sub>O-D<sub>2</sub>O exchange experiment have a materially different shape. Particularly overall intensity in the T<sub>2</sub> value range, marked as “core fluid signal” (< 100 ms) is smaller and the signal, which is due to H<sub>2</sub>O molecules dissolved in heavy water surrounding the core sample is higher at the end of the experiment in comparison with what is observed in the beginning of the experiment. The core fluid signal decreases linearly with the square root of time indicating that a one-dimensional diffusion process controls the transfer of H<sub>2</sub>O species from the core sample to the surrounding D<sub>2</sub>O liquid until it reaches the plateau. At this point the transfer of all H<sub>2</sub>O molecules that could be moved by isotopic exchange outside of the sample is finished. So, the difference between the core fluid portion of the measured NMR T<sub>2</sub> signal in the beginning and at the end of the H<sub>2</sub>O-D<sub>2</sub>O exchange experiment corresponds to the volume of water that can leave the core sample, and it should correspond to the upper level of BVW\_free parameter. The lower limit of bound water volume in this case is equal to BVW\_bound = BVW - BVW\_free, where BVW is derived from the volumetric core analysis data.

Because heavy water surrounding the core sample in the H<sub>2</sub>O-D<sub>2</sub>O exchange experiment does not contain any ions in parallel with H<sub>2</sub>O diffusion from the core sample, salt ions present in the water inside the core sample are also diffusing out of the core sample and into the heavy water surrounding it. The increase of the salinity of heavy water, surrounding the core sample, during the H<sub>2</sub>O-D<sub>2</sub>O exchange experiment results in a gradual decrease of the relaxation time for H<sub>2</sub>O molecules dissolved in D<sub>2</sub>O as it can be seen in Figure 10.

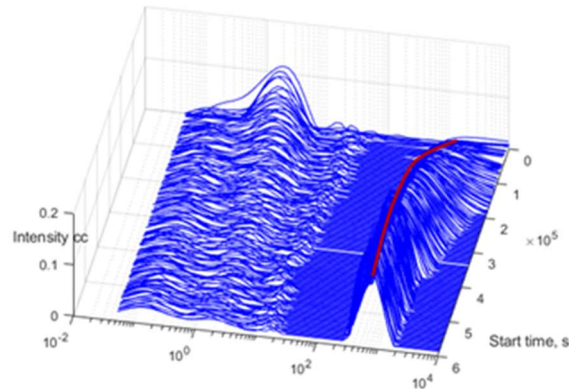


Figure 10: An evolution of the NMR T2 distribution during the H2O-D2O exchange experiment; the red line marks the position of the peak corresponding to the signal from H2O molecules dissolved in heavy water.

Eventually at the end of the H2O-D2O exchange experiment the amount of ions and H2O in heavy water should be very close to the amount of the same species originally present in the core sample if it can be assumed that the volume of D2O liquid surrounding the core sample is much bigger than the volume of native water inside the core sample. By measuring H2O volume and the amount of salt diffused out into the heavy water effluent an assessment of the formation water salinity can be performed. H2O volume can be quantified from NMR measurement performed on the post-experiment effluent. The amount of salt can be derived from effluent conductivity measurements. The cross plot of NaCl mass vs H2O volume derived for multiple samples run through the H2O-D2O exchange experiment allows to calculate formation water salinity (see Figure 11).

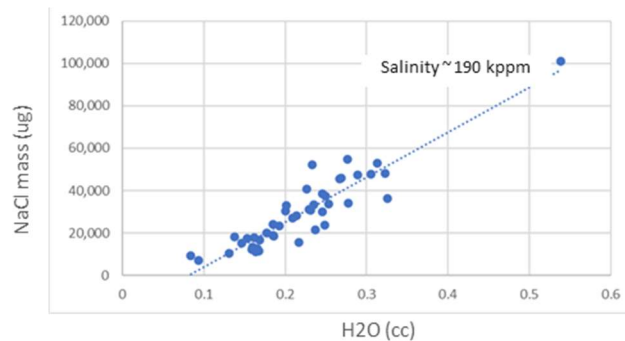


Figure 11: Results of the measurements of salt mass and H2O volume in H2O-D2O exchange experiment effluent performed for multiple Vac Muerta core samples; the slope of the fit defines the salinity of the native formation water.

The final part of the developed core analysis workflow is the permeability experiment. Figure 12 illustrates the design of the permeameter, which is used for the permeability experiment. It operates in the steady-state flow regime and uses upstream and downstream pumps run in the constant pressure mode, maintaining the constant pressure difference across the sample. Upstream and downstream flow rates are constantly monitored and if they are equal and steady-state flow regime is established, apparent permeability is calculated. During such an experiment sample pore pressure is maintained at a constant level and overburden pressure is increased in steps allowing to probe how sample flow properties change with increasing Vertical Effective Stress (VES). At the end of the experiment VES is decreased to become equal to its initial value to verify the presence and a degree of hysteresis in the behavior of apparent permeability.

Figure 13 shows an example of apparent permeability data acquired during the permeability experiment. For each VES step the apparent permeability decreases rapidly in the beginning and levels off closer to the end indicating sample compaction. Values of the apparent permeability at the end of each VES step

are assumed to be equal to the permeability defining the flow through the sample after an infinitely long exposure to such overburden pressure.  $K(VES)$  values derived from such a permeability experiment (see Figure 13) are used to calculate a value of the permeability for a reservoir in a “virgin” state  $K_0$  (at nominal VES value equal 0) and a value of a compaction factor  $\gamma$  by fitting  $K(VES)$  data with the formula  $K = K_0 * e^{-\gamma * VES}$ . Examples of  $K(VES)$  data sets and fits for samples with different degrees of compaction are shown in Figure 14.

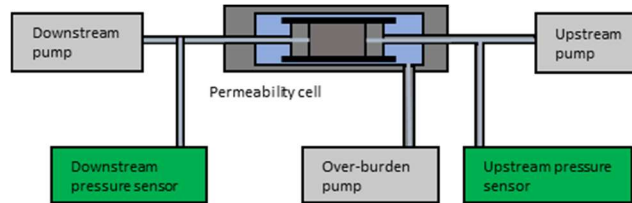


Figure 12: The scheme illustrating the design of the permeameter operating in the steady-state flow regime with upstream and downstream pumps operating in the constant pressure mode.

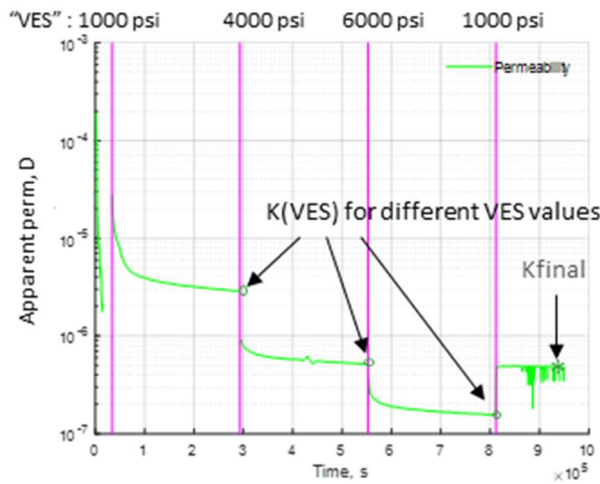


Figure 13: An example of apparent permeability data acquired during permeability experiment for an organic rich shale sample.

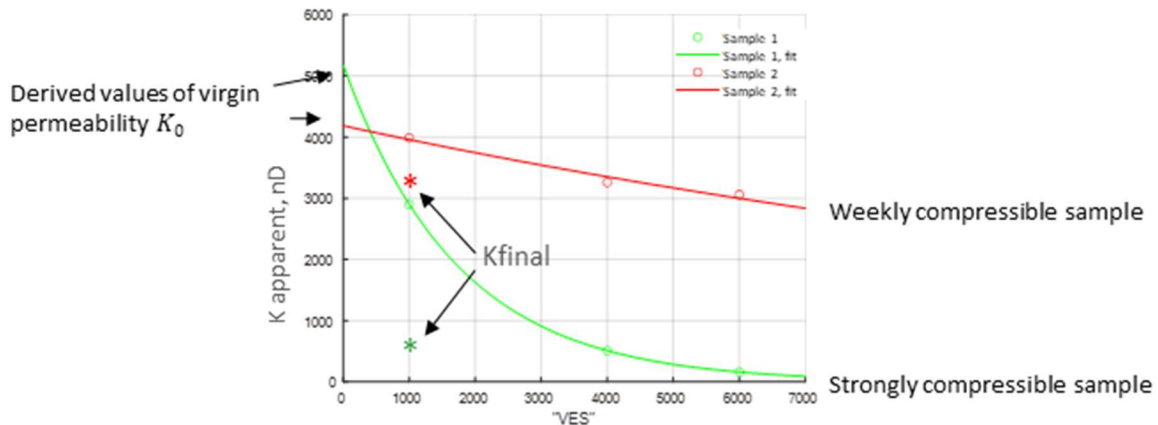


Figure 14: Results of the fitting of  $K(VES)$  data derived from permeability experiment for weakly compacting (red line) and strongly compacting (green line) samples of organic rich shale samples.

## Results

As mentioned above, core samples of organic rich shales lose significant portion of fluid occupying pore space *in situ* during core recovery because of HC fluid expansion caused by pressure decrease. As a

result, data acquired for as-received samples using the core analysis workflow described above do not provide a full picture of possible NMR responses for a particular formation. To overcome this challenge the pore space of the core samples needs to be re-filled with an NMR sensitive liquid such as H<sub>2</sub>O brine. To be able to monitor the evolution of the NMR signal during such a re-fill the Pressure Saturation Experiment (PSE) can be used. Figure 15 shows a scheme illustrating PSE.

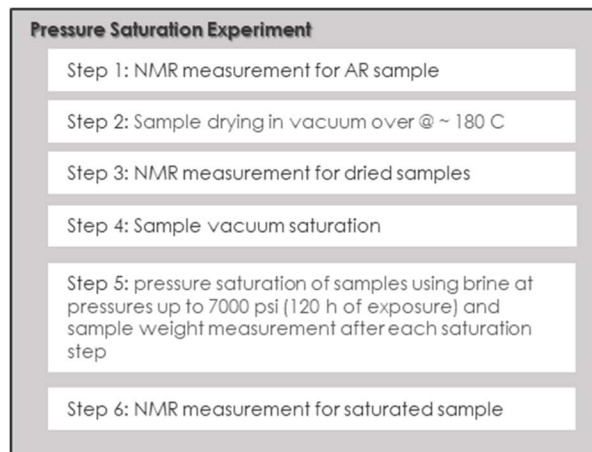


Figure 15: Scheme illustrating Pressure Saturation Experiment.

NMR data acquired at Step 1, Step 3 and Step 6 enable the calculation of NMR response of the fluids only present in the pore space for as-received samples and NMR response of the fluids only present in the pore space for saturated samples. Such differential NMR responses are derived by inverting the difference between an echotrain for an as-received sample and an echotrain for a dry sample (“AR – Dry” T<sub>2</sub> response) and by inverting the difference between an echotrain for saturated sample and an echotrain for dry sample (“Sat – Dry” T<sub>2</sub> response). Figure 16 shows examples of such T<sub>2</sub> responses obtained for a set of Vaca Muerta core samples, black oil maturity window, using PSE generated data and NNLS fit based inversion with Tikhonov regularization. Except for three components, identified in the NMR response for as-received samples before, two more components (Peak 4 and Peak 5) can be observed in “Sat – Dry” T<sub>2</sub> responses at longer T<sub>2</sub> values indicating that Peak 4 and Peak 5 correspond to fluids present in voids, whose characteristic dimensions are much bigger than the size of the biggest pores retaining fluids in the case of as-received samples. The analysis of the intensities of peaks extracted from “Sat – Dry” T<sub>2</sub> responses using FMFI (Nikitin et al 2017) indicates that the sum of the intensities of Peak 1 + Peak 2 + Peak 3 + Peak 4 correlates well with total porosity  $\Phi_{VAW}$  measured using the Volumetric Analysis part of the described above Core Analysis Workflow performed on twin plugs (see Figure 17). Such correlation indicates that pore volume corresponding to Peak 5 component was not present in as-received samples while the pore volume corresponding to Peak 4 was present but did not contain a material amount of fluid.

As it was discussed in Nikitin et al 2017 the concentration of residual fluid occupying a particular part of the porosity system in as-received samples of organic rich shales can be considered as volumes of fluid that are not going to be produced under any circumstances because it is impossible to bring the reservoir pressure to 1 atm. So, by comparing intensities of the corresponding NMR components for “AR – Dry” T<sub>2</sub> response and for “Sat – Dry” T<sub>2</sub> response the relative ability of fluids occupying each part of the porosity system can be estimated. This is particularly important for HC-occupied OM and matrix parts of the porosity system because such comparison allows to estimate relative producibility of the reservoir depending on its porosity structure ( $\Phi_{OM}$  vs  $\Phi_{matrix}$ ). Figure: 18 shows cross plots allowing to compare intensities of Peak 1, Peak 2 and Peak 3 components derived from “AR-dry” and “Sat-dry” T<sub>2</sub> responses using FMFI. It can be seen that pressure saturation managed to restore the intensity of Peak 1, which corresponds to BVW, to the level observed in as-received samples. The intensity of Peak 2 in

“Sat-dry” T2 response is 30 % higher than in “AR-dry” T2 response and Peak 3 intensity was increased by pressure saturation by 70 % in comparison with samples in as-received state. Taking into consideration that Peak 2 corresponds to the fluid in OM pores and Peak 3 corresponds to the fluid in matrix pores these results can be interpreted as the following: ~ 20 % of HC in OM pores and ~ 40 % of HC in matrix pores are expelled when pore pressure drops to 1 atm. This indicates that hydrocarbon fluid in matrix pores is ~ 2 times more producible in comparison with oil residing in organic matter pores for the Vaca Muerta formation, black oil maturity window.

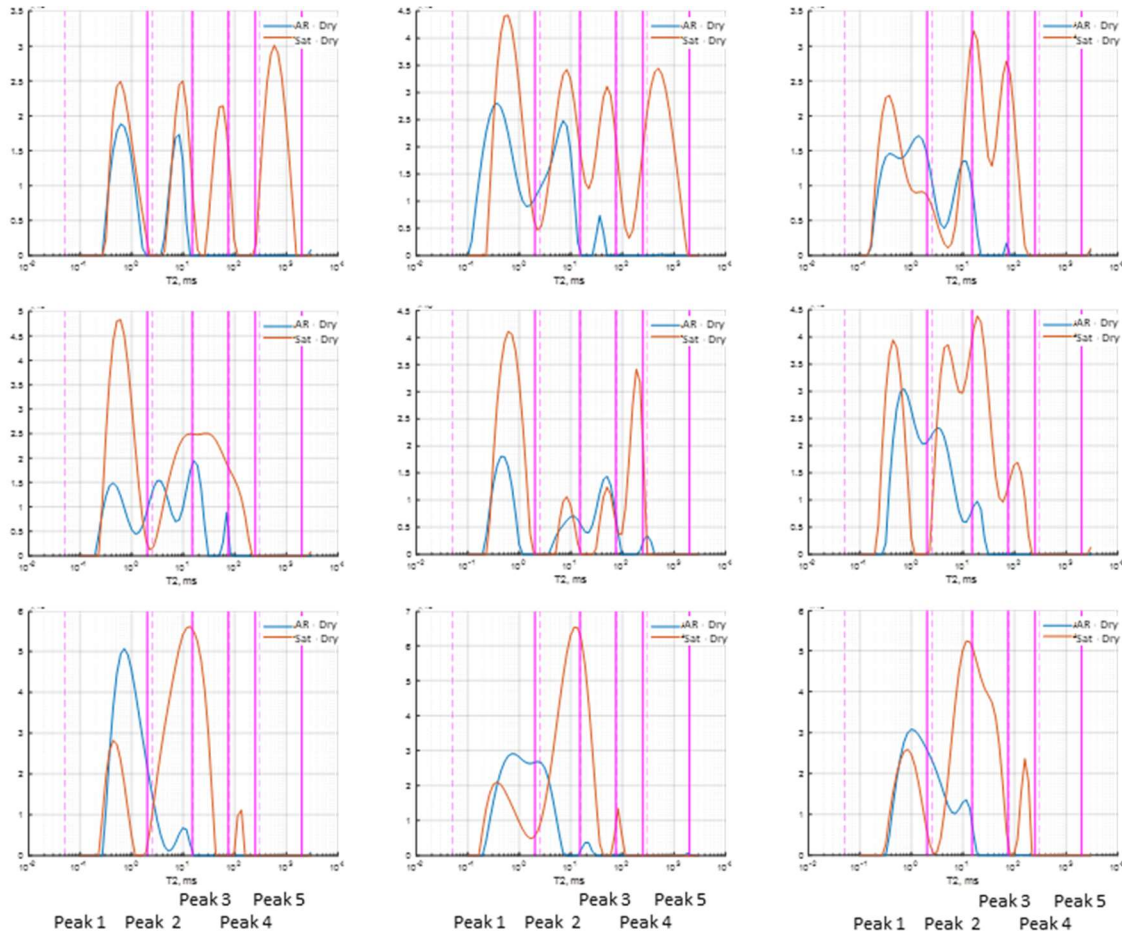


Figure 16: Examples of differential NMR T2 responses for a set of Vaca Muerta formation core samples; T2 distributions were obtained from echotrain differences using NNLS fit based inversion with Tikhonov regularization.

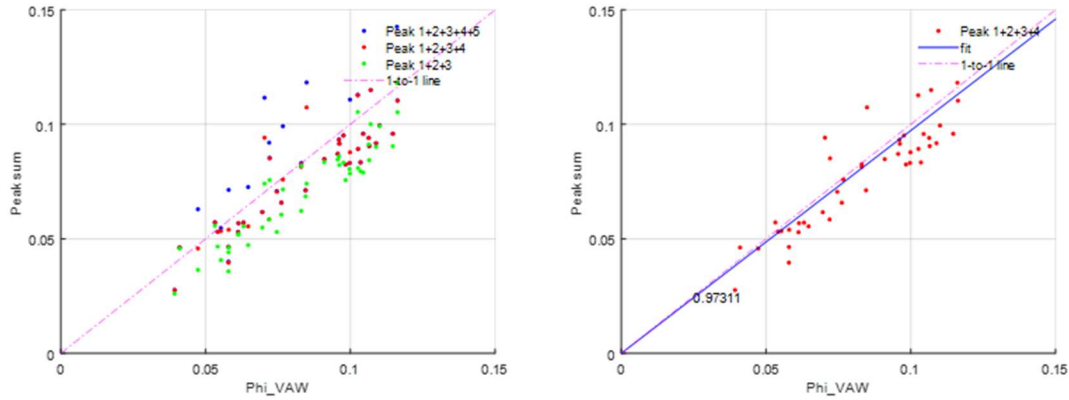


Figure 17 Comparison of sums of peaks derived from “Sat – Dry” T2 responses using FMBI with total porosity quantified using Volumetric Analysis Phi\_VAW.

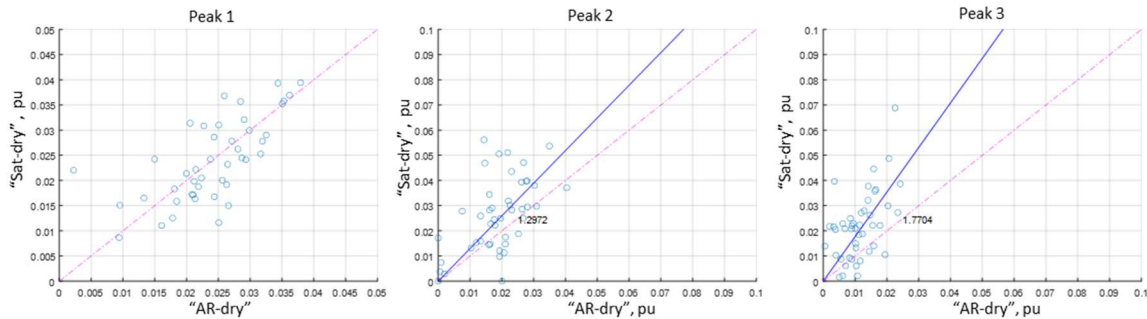


Figure: 18 Comparison of intensities of Peak 1, Peak 2 and Peak 3 components derived from “AR-dry” and “Sat-dry” T2 responses using FMBI.

To probe the connectivity of different parts of the porosity system of the Vaca Muerta formation permeability experiments described above were performed for a set of core samples that were used for Pressure Saturation Experiments. Before permeability measurements, the core samples were carefully dried in vacuum at less than 100 C till the weight stabilized and then saturated using HC liquid (dodecane) through a combination of vacuum and pressure saturation. Dodecane was chosen as the working liquid for permeability experiments because in the case of the Vaca Muerta formation the majority of the liquid flowing through the porosity system is oil. Figure 19 shows a typical response of the apparent permeability to VES changes derived from permeability experiment data for the Vaca Muerta formation core samples used in this study. One of the models that can be used to interpret such behavior, is based on the assumption that there are two flow systems, propagating through core samples. System 1 is much more permeable in a “virgin” state but also very compactable in an irreversible way. System 2 is much less permeable but also much less compactable. As a result, for the VES values up to around between 3000 psi and 4000 psi most of the flow happens through System 1 and at VES value around 4000 psi and above the flow through System 2 starts to dominate. Moreover, when the VES value returns to the value used in the beginning of the experiment liquid continues to flow through System 2 (System 1 is irreversibly closed by the sample compaction).  $K_0$  and  $\gamma$  parameters for each flow system are derived using red fit for System 1 and blue fit for System 2 (see Figure 19).

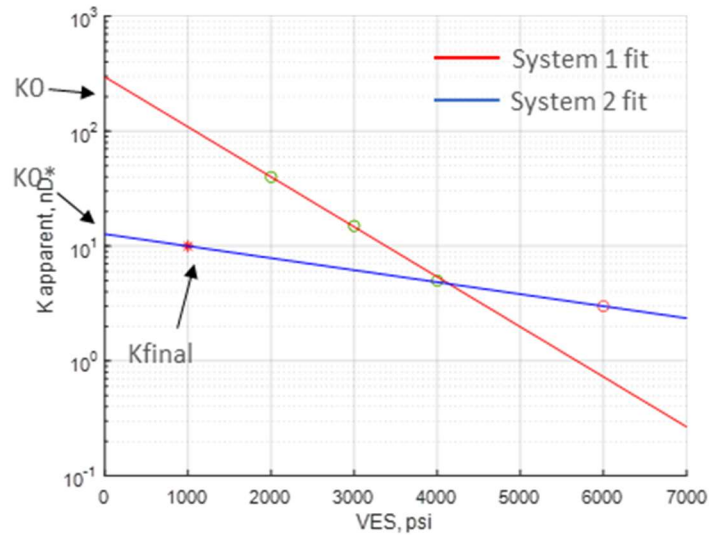


Figure 19: Typical response of the apparent permeability to VES changes derived from permeability experiment data acquired for Vaca Muerta core samples.

The analysis of the correlation of volumes of fluids occupying different parts of the core sample porosity system derived from PSE data (in the form of peak intensities) to  $K_0$  and  $K_0^*$  parameters derived from permeability experiment data was performed. It was found that there is a strong correlation between  $K_0$  and the sum of Peak 4 and Peak 5 and a correlation between  $\log(K_0^*)$  and the sum of Peak 2 and Peak 3 (see Figure 20).

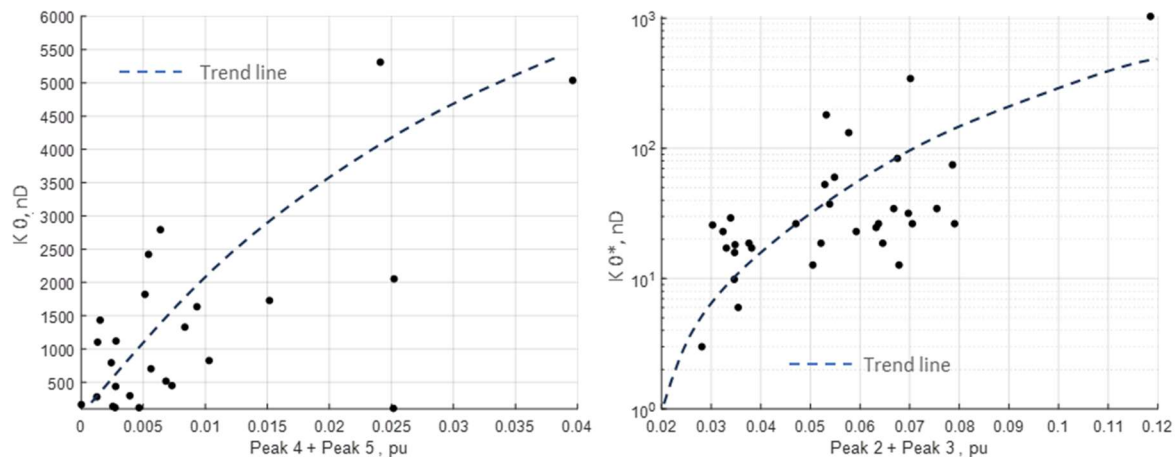


Figure 20: A cross-plot of  $K_0$  vs the sum of Peak 4 and Peak 5 (left) and a cross-plot of  $K_0^*$  vs the sum of Peak 2 and Peak 3 (right).

If there is a correlation between a permeability and the volume of fluid occupying a particular part of the porosity system in a linear-linear scale then from the flow properties point of view this part of the porosity system should have a minimal tortuosity. For example, a network of microfractures oriented mainly along bedding planes could have such  $K_0$  vs microfracture volume dependence. As discussed above, volumetric data analysis results indicate that Peak 5 is due to the fluids occupying microfractures induced in the samples. So Peak 4 could be due to another microfracture network that should be present *in situ* and could be formed due to the very high pore pressure of the Vaca Muerta formation.

The dependence of  $K_0^*$  on the sum of Peak 2 and Peak 3 in log – linear scale indicates that from a flow property point of view both organic matter porosity and matrix porosity operate as a single flow system, and they are well interconnected with each other.

So the analysis of PSE and permeability experiment data combined with the results of volumetric core analysis allows to derive the following interpretation scheme of NMR T2 distribution for the Vaca Muerta formation: Peak 1 is due to BVW and NMR signal from solid OM, Peak 2 is due to HC in OM network, Peak 3 is due to HC in matrix porosity and Peak 4 is due to HC in the system of microfractures present *in situ*. Such an NMR signal decomposition scheme can be applied to downhole NMR measurements, delivered by wireline NMR logging tools. Figure 21 shows NMR T2 distributions delivered by a vendor from the downhole data acquired in the well where a core was taken for this study. Analysis of the shapes of such T2 distributions allows to define boundaries for each peak position required by the FMBI.

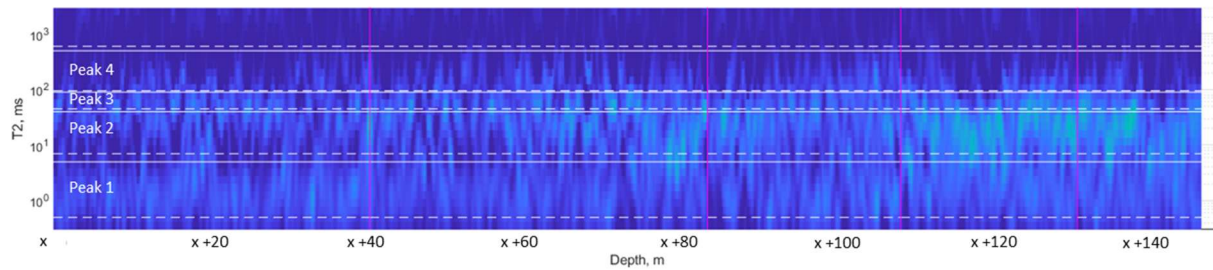


Figure 21: NMR T2 log delivered by the vendor from downhole NMR measurements and T2 boundaries for the peak positions for Forward Modelling Based Inversion (white lines).

FMBI was applied to the echotrails acquired by the downhole NMR tool and intensities of all peaks constituting *in situ* NMR response of the Vaca Muerta formation were extracted. Figure 22 shows the comparison of intensities of different peaks derived from the core PSE data and the corresponding downhole NMR measurements. A reasonable correlation for Peak 2 and Peak 3 can be seen in the left cross-plot taking into consideration the heterogeneity of the Vaca Muerta formation and the vertical resolution of the NMR logging tool used to acquire downhole NMR data. For Peak 4, such a correlation is weaker (the right cross-plot) but mainly because of the  $\sim 4$  times lower Peak 4 intensity ( $\sim 0.0075$  pu on average), which is comparable with the uncertainty of downhole NMR measurements.

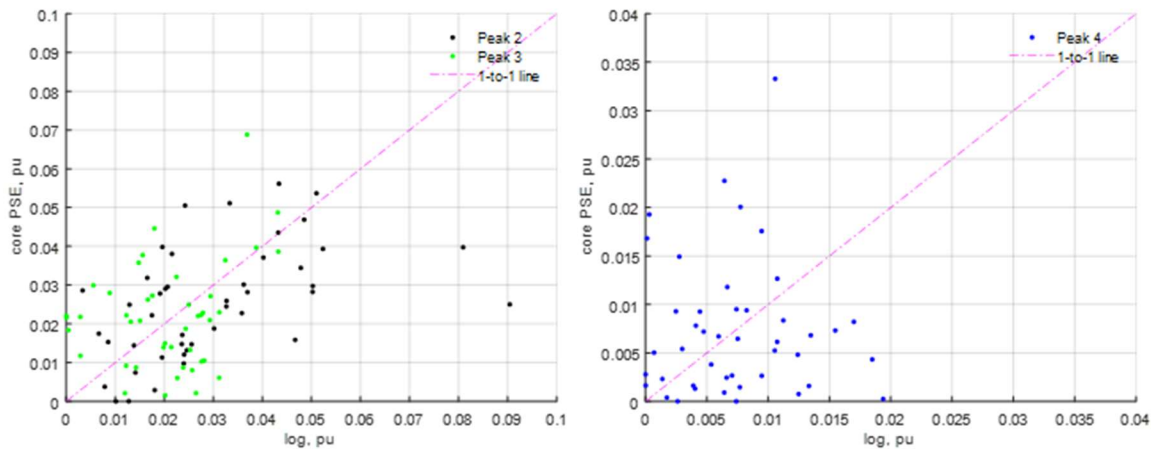


Figure 22: Comparison of the intensities of different peaks derived from the core PSE data and corresponding downhole NMR measurements; Peak 2 and Peak 3 (left) and Peak 4 (right).

Figure 23 shows the sum of Peak 2 and Peak 3 and Peak 4 derived from the NMR log using FMBI. This shows that the total volume of HC residing in the porosity system ( $\Phi_{OM} + BVHC_M$ ) increases with the depth significantly which is mainly driven by the increase of OM concentration with depth for this well. At the same time the concentration of HC in microfractures does not change with depth significantly indicating that the assumption that the microfracture network is controlled by the local pore pressure discussed above could be correct.

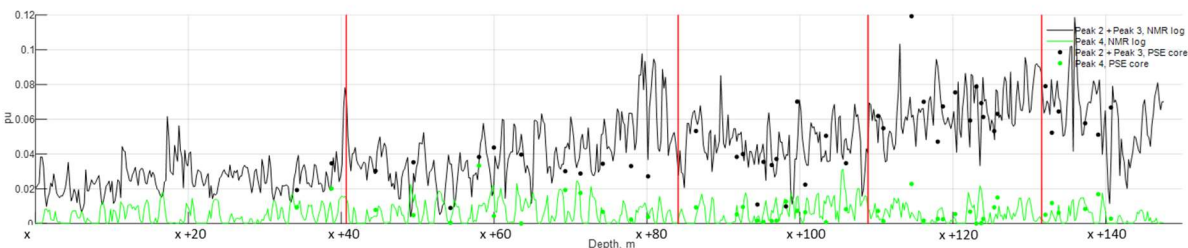


Figure 23 The sum of Peak 2 and Peak 3 and Peak 4 derived from downhole NMR measurements using FMBI and comparison with corresponding core data

Peak 1 derived from the downhole NMR measurements consists of both water and solid OM NMR signals. If the concentration of OM is available from downhole geochemical measurements (derived from the results of induced gamma ray spectroscopy measurements) then a rough correction could be applied to Peak 1, so that the impact of solid OM NMR signal could be minimized. Such a correction is based on the subtraction of TOC derived from a geochemical log multiplied by a correction factor. The value of the correction factor  $\alpha$  is derived from the correlation of BVW values measured for core samples and Peak 1 -  $\alpha \cdot \text{TOC}$  from the downhole measurements where  $\alpha$  value is chosen to provide the best match of core BVW and TOC corrected Peak 1 from the logs. Cross-plots of core BVW values vs corresponding values of Peak 1 from the log without OM correction and with OM correction are shown in Figure 24. Figure 25 shows a comparison of the intensity of Peak 1 derived from the NMR log using FMBI and corrected for solid OM NMR signal, using TOC geochemical log, and corresponding BVW data derived from core measurements. A good match is observed between core and log data, which demonstrates that such a simple linear correction allows to extract BVW from the combination of NMR and geochemical logs. Except BVW data points, derived from core measurements (blue dots), also BVW\_bound data derived from H<sub>2</sub>O-D<sub>2</sub>O exchange experiments (blue circles) are shown in the same plot (see Figure 25) It can be seen that the ratio of BVW\_bound and BVW varies significantly with depth and additional analysis is required to establish which sub-surface parameters control such ratio.

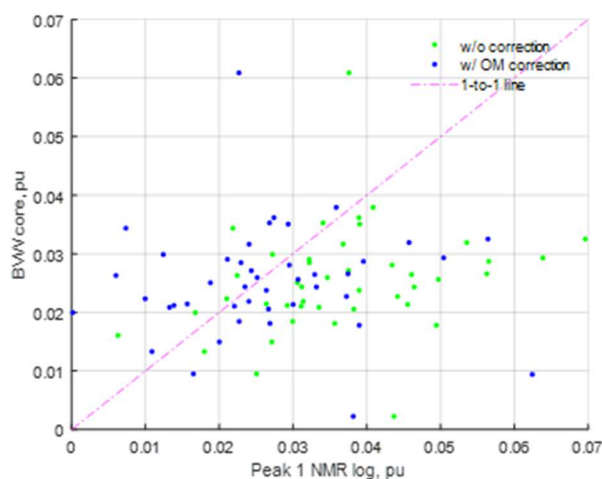


Figure 24: Cross-plots of core BVW values and Peak 1 from NMR log from corresponding depths without OM correction and with OM correction from TOC log delivered by geochemical logging tool.

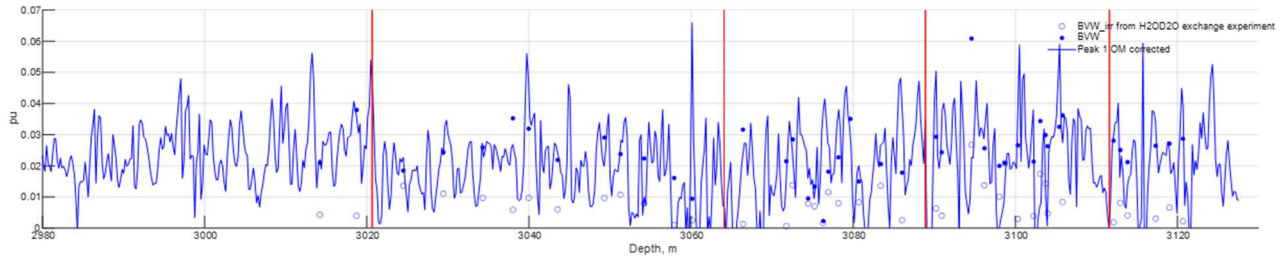


Figure 25: Intensity of Peak 1 derived from downhole NMR measurements using FMBI and corrected for solid OM NMR signal using TOC geochemical log and comparison with corresponding BVW and BVW\_bound data derived from core measurements.

So, the results of the study presented in this paper allow to evaluate advanced petrophysical properties of the Vaca Muerta formation if NMR and geochemical logs are available. In the case where such logs have not been acquired and only a standard logging tool suite was run, additional efforts have to be spent to develop an interpretation scheme capable of extracting advanced petrophysical parameters from logs like resistivity, gamma ray, density, and neutron porosity. A workflow that could allow to develop such an interpretation scheme is illustrated in Figure 26. In this workflow core data are used to develop NMR log interpretation scheme that is applied to a set of wells where both advanced and standard logs are available. The next step is to use continuous advanced petrophysics property curves, extracted from NMR logs, as data sets used for the calibration of the interpretation scheme of interest.

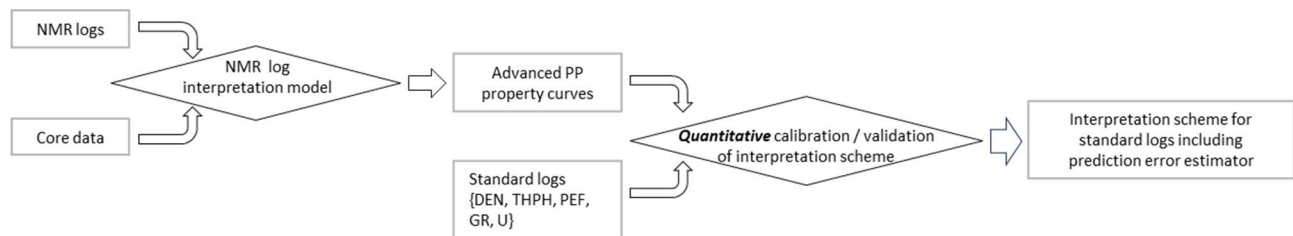


Figure 26: Scheme illustrating the workflow which could be used to develop an interpretation scheme for standard logs allowing to evaluate advanced petrophysical properties of Vaca Muerta formation.

There are several advantages to this approach compared to the classical one, where the standard log interpretation scheme is derived using core data. Particularly, the proposed approach compares data from NMR logs with data from the standard logs, which are recorded at the same scale and thus the upscaling issue has a much smaller effect on the results. Also, it makes available a large number of data points (from 1000 to 10000) for interpretation scheme development enabling the use of machine learning based algorithms for quantitative calibration of such schemes. Such statistically large data sets are also suitable for the development of error estimators capable of providing P10, P50 and P90 estimates for advanced petrophysical properties of interest which should facilitate better business decisions.

## Conclusions

In this paper, it is demonstrated how advanced NMR measurements, in combination with other core analysis techniques, can be used to develop an NMR data interpretation scheme for the Vaca Muerta formation, in the black oil maturity window, allowing for the quantification of BVW, BVHC\_OM and BVHC\_M from downhole NMR measurements. Also, the analysis of the data acquired for a large set of plugs run through permeability and pressure saturation experiments indicates the presence of a microfracture network *in situ* filled with oil whose concentration can also be derived from the NMR T2 response, both for core and downhole measurements, using Forward Modelling Based Inversion. Advanced volumetric parameters derived from NMR T2 data can also be used to assess the flow properties of the formation porosity system and microfracture network using results of permeability experiments. H2O-D2O exchange experiments systematically performed for as-received core samples allowed to estimate formation water salinity to be equal to 190 kppm, and the volumes of bound water,

BVW\_bound, were quantified. The analysis of Pressure Saturation Experiment data allowed for the conclusion that HC occupying the matrix part of the porosity system are 2 times more producible than HC occupying the OM porosity.

### **Acknowledgments**

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