Determination of clay-bound water in non-conventional oil reservoirs via T_1 NMR measurements. A case study for the Vaca Muerta formation.

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Abstract. Laboratory ^{1}H nuclear magnetic resonance (NMR) is a fast, reliable, and non-destructive method that is widely adopted in the oil industry. It allows the quantification and typification of organic solid matter and fluids in source rocks. Among the many constituents present in oil and gas reservoirs, the clay matrix content has been identified as a key contributing factor for reservoir quality evaluation. Correct quantification of clay bound water (CBW) gives insight on clay content and allows the determination of effective porosity. In conventional rock samples, 1D T_2 measurements may be used to quantify CBW. In unconventional rocks the preferred methodology is a time consuming 2D T_1-T_2 relaxation map acquisition. In search of reducing measurement time, 1D T_1 measurements are analyzed to derive a quantitative value of CBW. We aim to compare the different approaches taken to obtain CBW values and understand advantages and limitations of each method. 1D NMR results are directly compared to 2D NMR to validate the validity of each approach. Furthermore, clay content from XRD experiments is used to correlate measured CBW values. Results from 3 different wells from the Vaca Muerta Formation in the Neuquén basin of Argentina are shown.

1 Introduction

The oil and gas industry has always relied on laboratory characterization of rock samples to shine light on the physical and chemical processes occurring in depth. Although there is a wide variety of logging tools available, well profile models are calibrated and verified using precise and punctual data points obtained from a detailed characterization of available rock samples. Well logging allows for a continuous measurement inside the well but has limited vertical resolution. Well rock samples give very precise and punctual values, but the construction of a continuous profile requires the extraction and measurement of a large number of samples, which translates to time and money. A balance between both must be achieved.

In the present context, there is a need for the development of new and efficient laboratory workflows focused on rock characterization. In the present work we focus our attention on petrophysical properties, with special focus on the quantification of CBW.

NMR is a standard technique used both in well logging tools and laboratory analysis. For conventional reservoirs several protocols have been developed through the years, however, a straightforward implementation to non-conventional reservoirs is not possible. The main reason is that conventional reservoirs, mainly composed of sand packing of chalk, are silent to NMR and only the fluids present in the porous system are detected. Non-conventional reservoirs are characterized by the presence of organic matter, kerogen or bitumen, which introduces organic porosity were hydrocarbons are confined in nanopores. This leads to a complexity of the relaxation times of the signals of the different detectable components: organic matter, hydrocarbons in organic pores, hydrocarbons and water in inorganic pores, clay-bound water (CBW) that in general require 2D methods to resolve the different contributions [1-4]. These measurements are time-consuming, and the deconvolution methods are still dependent on the operator skill.

At Y-TEC we have developed an in-house workflow to measure unconventional rock samples “as-received”, using NMR to characterize the fluid components that remain in the rock [5]. This workflow includes 1D and 2D measurements, for which the sample must remain in the NMR core-holder for a considerable amount of time (dependent on the amount of fluid present in the rock). The T_1-T_2 maps allow for fluid typing and effective porosity determination.

The issue with this experimental methodology arises when preserved samples are to be measured before water saturation measurements. Once these samples are

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unpreserved, manipulation time must be minimized to prevent the excessive loss of fluids. Thus, 2D NMR measurements are usually avoided since they are time-consuming [6]. This motivates the need for exploring different techniques to correctly quantify CBW, with time reduction being a key factor.

In conventional rock samples, a good agreement between low field NMR T_1 and T_2 distributions can be seen. This allows the use of T_1 or T_2 distributions to separate CBW from fluid in larger pores [7]. Straley et. al. used T_1 distributions with cutoff times to separate irreducible water (CBW and capillary-bound water) [8]. But this was done on sandstone samples which are not as complex as shale samples. In 2016 Yang and Kausik proposed a T1 cutoff to separate CBW from other components such as bitumen and kerogen components but in NMR T1-T2 maps at 400 MHz [9].

In this work we aim to compare the use of different NMR laboratory experiments to correctly quantify CBW from unconventional reservoir rocks. By using previous acquired knowledge on 2D NMR T_1-T_2 maps on rock samples from the Vaca Muerta formation, we can understand where the signals of different fluid components inside the rock appear. This insight allows us to explore the use of 1D NMR distributions to quantify components such as CBW.

Samples from 3 different wells from the Vaca Muerta formation were measured. Validation of the proposed methodology is performed on one of the wells and then tested on two wells using well logging data to compare results obtained.

2 Experimental Methodology

For conventional rock samples NMR data analysis is much more straightforward and the use of 1D NMR distributions is a well-established practice. The use of cutoff times is accepted in the community and allows for the identification and quantification of different fluids present in the rock. NMR T_1-T_2 relaxation maps are mainly used to separate signals from different components that overlap in a 1D measurement.

Fluid typification in rock samples from NMR 2D maps has been widely studied at different magnetic fields (Larmor frequency) [10-13]. It is well established that different components present in rock samples share similar T1 or T2 decay times, and thus 2D maps must be measured to correctly separate and quantify them.

In figure 1 we show a T_1-T_2 NMR map for a shale rock sample measured using an Oxford Instruments Geospec 2 analyzer at 2.27 MHz. Three main fluid components can be discriminated: hydrocarbon in organic pores (OP), clay-bound water (CBW) and fluids in inorganic pores (IP) including water and hydrocarbons [4]. From this map we can see that the signals from CBW and hydrocarbons in organic porosity have very similar T_2 decay times. A similar overlap is observed in T_1 distribution measurements, where hydrocarbons in organic pores have the same decay time as fluid in inorganic pores. When time is not an issue, measurement of a T_1-T_2 NMR map is the ideal methodology for fluid typing and quantification.

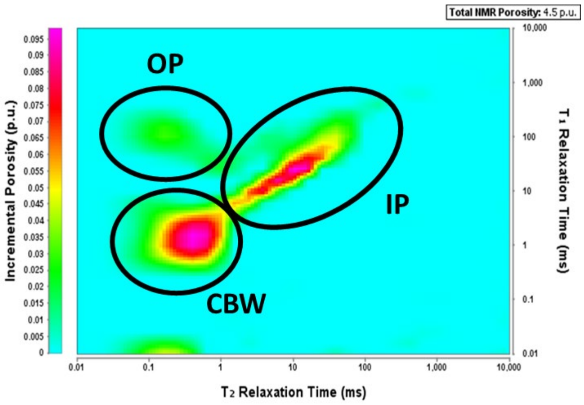

Fig. 1. T_1-T_2 NMR map of a source rock. Three regions of the map are marked which correspond to organic pores (OP), clay bound water (CBW) and inorganic pores (IP).

There is an alternate method for fluid typing based on 1D measurements that reduces measurement time by a factor of 2 (at same SNR). This methodology uses only T_1 and T_2 distributions.

T_1 and T_2 distributions are merely the collapse of a T_1-T_2 relaxation map in the vertical and horizontal directions, respectively. Therefore, in the T_1 distribution, OP and IP will be indistinguishable, but CBW can be separated as the short T_1 time component. In the case of the T_2 distribution, OP and CBW will overlap in time, but IP can be separated as the long T_2 component. Therefore, using both T_1 and T_2 distributions, all components can be obtained.

A simple and fast approach to separate components using 1D distributions would be the use of T_1 and T_2 cutoff times [8,9]. This is usually done in the industry and can lead to good results when components are clearly separated as shown in figure 2A. In non-conventional reservoirs this is not always the case due to the short relaxation times involved and commonly broad distributions, so we implemented a fitting of the relaxation times distribution, where the superposition of two log-normal distributions function proved accurate enough to quantify the different components.

A curve fitting script in Python was implemented. In figure 2B we show an example of an ill-posed distribution where the choice of a T_1 or T_2 cutoff does not suffice to separate the different contributions but is well represented by two log-normal distributions.

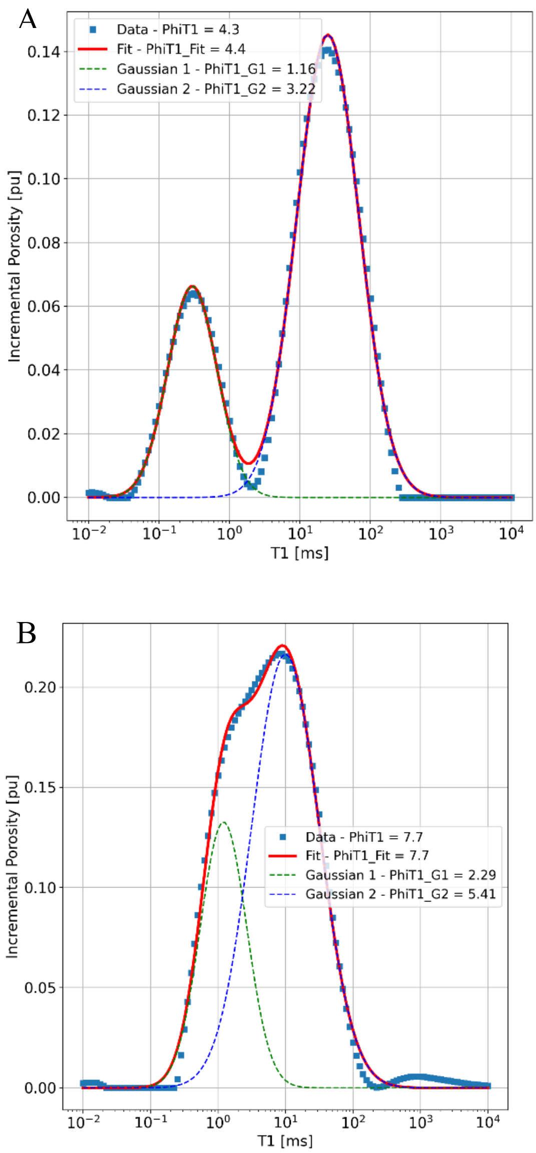

Fig. 2. Two 1D T_1 distributions. A. An example of two well defined and separated distributions where a T_1 cutoff would suffice to separate components. B. A clear example where a T_1 cutoff would be inadequate to characterize the contribution of each distribution.

Since our main goal is to quantify CBW using 1D distributions we will center our focus on T_1 distributions, where the component with low T_1 corresponds to CBW. It is important to point out that in an ideal scenario, measuring a T_1-T_2 map is always the preferred way to separate components inside the rock. However, when analyzing preserved samples, time is of the essence and T_1-T_2 maps take longer than 1D distributions. A longer measurement time can translate to a greater fluid loss in the sample, compromising further fluid saturation experiments. Therefore, having an experimental methodology that allows CBW determination at half the experimental time gains relevance.

The time difference between a T_1 distribution and a T_1-T_2 map with same SNR was shown to be around a factor of 2. This difference originates from the fact that in a T_1-T_2 map the first data point is acquired at the top of each spin-echo of the CPMG pulse sequence, whilst in a T1 distribution the first data point is measured right after a 90° pulse (FID sequence). This translates into having a larger initial signal to achieve the desired SNR (and thus fewer number of scans), added to the time difference between measuring a complete CPMG and an FID.

To test the proposed methodology samples from 3 different wells were measured: Well A in the wet gas window (15 sidewall rotary cores); Well B and C, both from the same play, in the oil window (48 and 27 sidewall rotary cores, respectively).

3 Results

The NMR measurements were performed using an Oxford Geospec2 analyzer at 2.27 MHz. T_1 distributions were measured using 30 time-steps and a recycle delay of 500 ms. The SNR was set above 100, which in consequence varied the number of scans and time between samples depending on the amount of hydrogen rich components inside the sample. For Well A, T_1-T_2 maps were also measured and CBW was quantified for each sample by segmenting the corresponding signal (see figure 1). In the case of Wells B and C, only T_1 and T_2 distributions were measured and thus a comparison of the methodology was done against NMR well logging profiles. For samples of all three wells, XRD mineral composition analysis was performed, and clay content was determined.

Clay content from XRD can be compared with CBW associated porosity to see if a good correlation is observed. But it is important to consider that the NMR measurement is performed over the whole sample, whilst XRD is measured on a few grams of rock and thus heterogeneity may have a large impact. Also, different clays can have different water storage and should also be studied. This is why these correlations are performed mostly to evaluate if there is a trend, but certain dispersion in the data points is expected.

The most straightforward comparison to validate the methodology is comparing T_1 CBW to T_1-T_2 CBW. In figure 3 we show the correlation for Well A, where the red solid line is the least-square fit of the data, and the green solid line is the linear function $x = y$.

T_1 distribution is overestimating the value of CBW by 13% and there is slight offset in the data. Nevertheless, the values align and show a very good correlation ($R^2 = 0.9577$). The difference between values may possibly be attributed to solid-like components in the rock with short T_1 values (OH present in clays and kerogen) that have not fully decayed before measuring the first data point of the FID [12]. The results show that the method is not perfect, but the differences are more than acceptable.

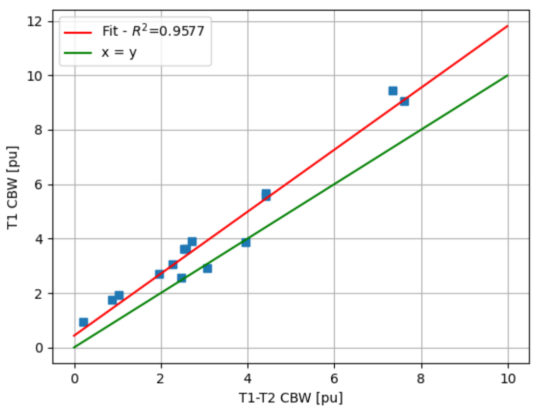

Fig. 3. Correlation of T_1 CBW versus T_1-T_2 CBW for Well A. In red a linear fit of the data points. In green the identity function.

Next, we compare values of both T_1 CBW and T_1-T_2 CBW with clay content from XRD mineral composition (see figure 4). We can see that both CBW have a linear correlation with clay content, but dispersion is greater in the case of the T_1 distribution. Once again, it is important to point out that dispersion may be associated to sample heterogeneity. NMR measurements average out differences in composition at the whole rock scale, while XRD measurements are performed on a subsample that may not be representative. Thus, these linear plots may be useful to spot heterogeneity of the sample and motivate more exhaustive procedures to obtain a representative subsample for XRD.

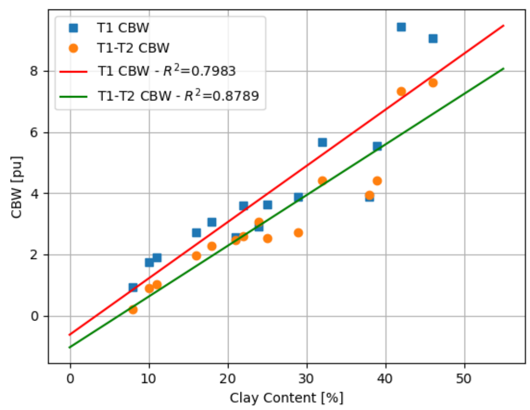

Fig. 4. CBW determined from T_1 distribution and T_1-T_2 maps as a function of XRD clay content. Linear fit for each dataset is shown in red for T_1 CBW and green for T_1-T_2 CBW.

For Wells B and C, no T_1-T_2 CBW laboratory measurements were performed. Therefore, CBW determination was done using T_1 distributions fitted with two log-normal distributions. To analyze the T_1 CBW we rely on XRD analysis and NMR well logging profiles. A few remarks regarding this comparison must be made clear beforehand. First, laboratory and well measurements are not performed on the same sample but are plotted at the same depth. This translates into accepting dispersion between data sets due to heterogeneities within the rock. Also, the well logging tool has a vertical resolution of around 30 cm and has a certain penetration length, whilst measurements were performed on sidewall rotary cores (1 inch diameter and 2 inch long). When petrophysicists interpret and compare laboratory results to well logs, they look for long scale fluctuations and that along the well the data points follow the same tendency as logging data.

Figures 5 and 6 show two different profile tracks for Wells B and C respectively. The tracks shown correspond to total porosity (left) and CBW (right) obtained using an NMR well logging tool. Data points measured in the laboratory were added to the plots: blue dots correspond to total porosity; green dots correspond to T_1 CBW.

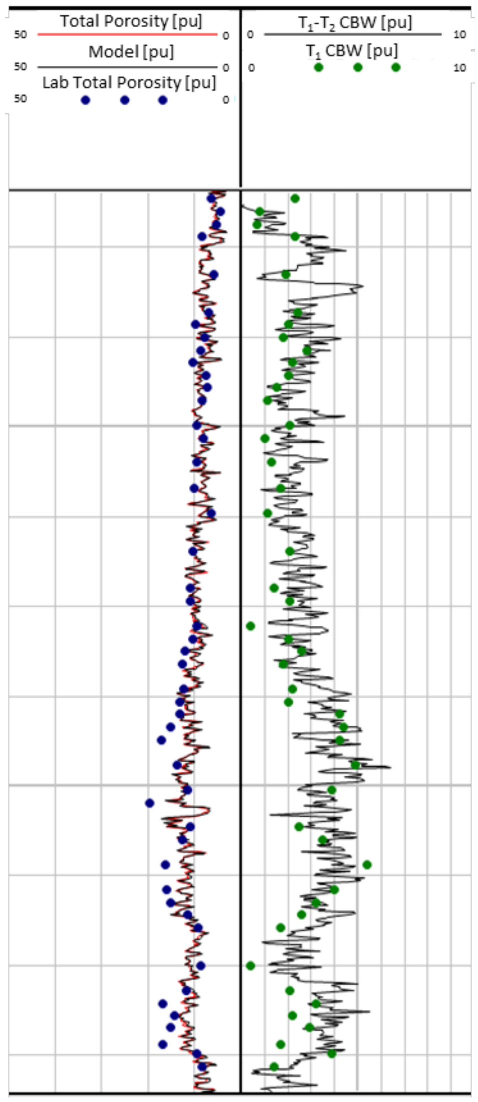

Fig. 5. Total porosity and CBW depth profiles for Well B. Laboratory petrophysical measurements on sidewall cores are plotted alongside well logging data.

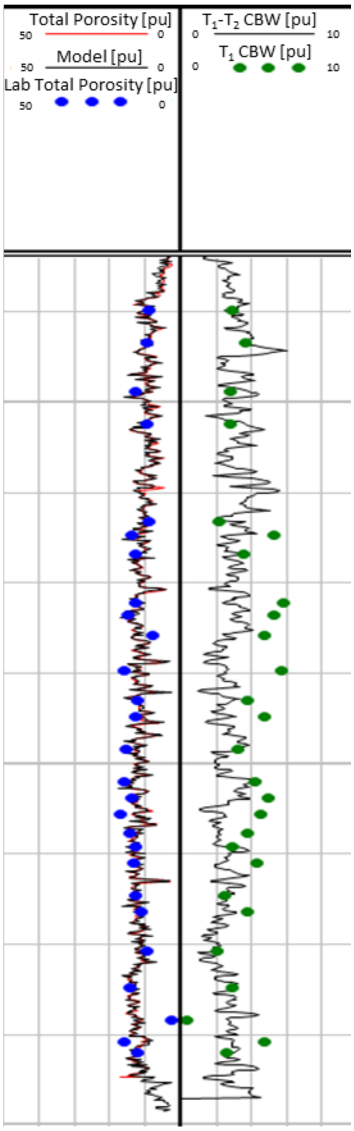

Fig. 6. Total porosity and CBW depth profiles for Well C. Laboratory petrophysical measurements on sidewall cores are plotted alongside well logging data.

A detailed comparison of well log values and laboratory values is sometimes quite difficult since measurements are not being performed on the same exct samples and thus lithological fluctuations and heterogeneity plays a significant role. This is why comparison is usually done on a large scale, looking at a general trend in values. In Well B T_1 CBW follows the trend of CBW measured inside the well, although a slight difference is observed in a few points deeper inside the well. The difference in values of CBW a standard deviation of 1.5 pu and a maximum difference of 2.6 pu.

In Well C, T_1 CBW values seem to overestimate the CBW measured inside the well, but the difference in values has a standard deviation of 1.5 pu and a maximum of 3 pu. These results still look promising considering the clarifications made beforehand regarding the differences in measurements.

Lastly, XRD measurements on samples were plotted against T_1 CBW for both wells B and C (see figures 7 and 8, respectively). These plots show a large dispersion that is most probably due to two main factors: sample heterogeneity and clay type. In the case of Well B, the dominant clay is illite-smectite with over 80% illite. This means that the water volumen associated with clay should not be a factor and the dispersion is most likely to be due to heterogeneity of the rock.

For Well C, XRD data shows mainly illite-smectite (80 % illite) and chlorite as the main clays. The presence of some samples with more chlorite than others may explain some of the dispersion observed. This is part of an ongoing study. Nevertheless, heterogeneity of clay distribution within the sample also plays a key factor.

The dispersion observed in the correlation with XRD data also supports the explanation given for fluctuations observed between well logs and laboratory data.

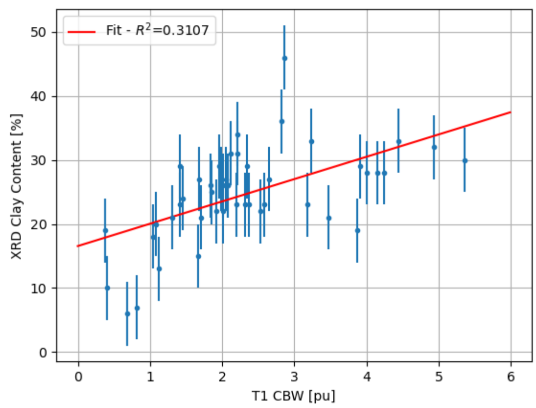

Fig. 7. XRD clay content as a function of T_1 CBW for Well B. A linear correlation of the data is sown (red solid line).

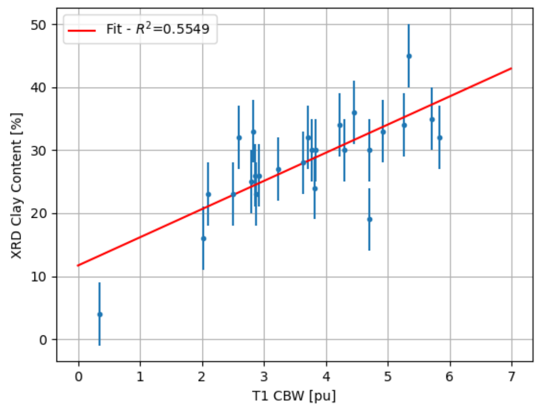

Fig. 8. XRD clay content as a function of T_1 CBW for Well C. A linear correlation of the data is sown (red solid line).

4 Summary and Outlook

In this work we presented the possibility of the determination of CBW from 1D T1 measurements. As 1D measurements are in general less time consuming than 2D ones, the aim is to reduce the time that a preserved core sample is under study. Even though relaxation maps have a high resolution in the determination of different components, whilst part of this information is collapsed in a 1D experiment, we found that in general a good correlation between the 1D measurements is obtained with 2D experiments, XRD determination of clay content and well logging essays. The method was implemented in a standard commercial software, and we envision that the method can be improved by the addition of a relaxation filter before acquisition of the data for the T1 determination, aimed to filter short-lived signals corresponding to bitumen for instance.

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