ULTRA FAST CAPILARY PRESSURE AND RESISTIVITY INDEX MEASUREMENTS (UFPCRI) COMBINING CENTRIFUGATION, NMR IMAGING, AND RESISTITY PROFILING

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ABSTRACT

The knowledge of water saturation is essential to determine hydrocarbon in place in an early discovery and also to monitor the sweep efficiency during the production phase. Water saturation can be measured using different logging techniques such as: resistivity, NMR (Nuclear Magnetic Resonance), dielectric, and pulsed neutrons. However resistivity measurement is the only means capable of giving information in the virgin zone around the well. These electrical measurements are used to estimate the water saturation using Archie's law.

Archie's law requires the knowledge of certain parameters such as the cementation factor (m) and the saturation exponent (n). These parameters can be measured in the laboratory and the time required to obtain these values differs considerably. While the cementation factor can be measured in hours, the saturation exponent measurement may take up to six months and therefore the cost associated to the experiment is higher.

The common technique to derive the saturation exponent is based on the use of a porous plate to ensure the uniformity of the saturation profile along the rock sample, but the price to pay is a very long and expensive experiment, driven by the low permeability of the porous plate.

This paper presents a new and radically faster alternative approach to measure both capillary pressure and saturation exponent. The general idea is to remove the porous plate and to generate a saturation profile in the sample using centrifugation. The generated saturation profile is then characterized by NMR and a multiple electrode resistivity profiling method developed "in house". This method can be applied together with the GIT (Green Imaging Technologies) patented capillary pressure method and therefore provide capillary pressure and resistivity index values in parallel.

This method has been tested and validated on outcrop samples from Bentheimer (sandstone) and Estaillades (carbonate). Also, it has been successfully applied to reservoir samples from a gas field under drainage and imbibitions processes.

INTRODUCTION

Accurate evaluation of hydrocarbon in place requires the knowledge of rock volume, porosity and fluid saturations. Resistivity measurements, performed during logging

operations, are converted into saturations using Archie's equation [1]. Thus, the knowledge of the Archie parameters "m" and "n" is essential. The cementation exponent "m" is easily and quickly determined in the laboratory from electrical measurements performed on 100% water saturated samples. In contrast, classical measurements of the Archie saturation exponent "n" are long and prone to experimental artifacts. The classical method relies on the establishment of a homogeneous water saturation profile along the sample using a porous plate, but this process can be extremely long. It may take from weeks (continuous injection method- no capillary equilibrium) to months (classical porous-plate method). Often, the results of laboratory measurements of the factor "n" arrive too late to be used in the electrical logs interpretation.

Attempts have been made in the past to shorten this experiment. Fleury proposed the FRIM method [2], which is much faster, but does not allow for simultaneous measurement of capillary pressure. Bona *et al.* [3] proposed a method based on centrifuge and saturation profiling. Assuming the validity of Archie's law, they calculate a saturation exponent "n".

Another drawback of the classical methods (porous plate or continuous injection) is that they are very sensitive to the heterogeneity of water saturation (Sw) in the sample. The only way to circumvent this issue is to use in-situ saturation monitoring techniques: Bona *et al.* [4] proposed to use MRI; Durand [5] proposed X-ray CT together with a first attempt to introduce resistivity profiling.

The method proposed in this paper combines centrifugation, NMR profiling and local resistivity measurements to derive the capillary pressure and the saturation exponent. It consists in generating a saturation profile along the sample by centrifugation at a given speed. Then, NMR is used to measure the saturation profile and resistivity is measured using an "in house" multi electrode device. This process is performed at different speeds in order to cover different ranges of capillary pressures and saturations. Results are available in weeks and the method is applicable to heterogeneous samples.

OUTLINE OF THE METHOD

This method starts with samples saturated 100% by brine, measuring NMR and resistivity profiles. Next, samples were centrifuged. This creates a saturation profile along the plug induced by the capillary pressure gradient applied in the centrifuge. NMR and resistivity profiles are measured again, and then converted respectively to saturation and resistivity index profiles using the data acquired at 100% water.

The resolution of the resistivity profile is limited by the electrode spacing, creating N virtual slices (for N+1 electrodes). Matching the saturation to the resistivity in each slice, N points are obtained in the Rt=f(Sw) plot at each centrifuge step.

An example is given on Figure 1 on which 7 electrodes were used (6 slices), Following this method, we obtain for each of the 6 slices a triplet [Pc, Sw, RI]; and therefore are able to plot the RI=f(Sw) curve together with the Pc=f(Sw) curve (as already proposed by Green *et al.*[6][7].

Minimal redistribution of fluids was observed during the timescale of NMR and resistivity measurements, and affected neither NMR nor resistivity profiles.

EQUIPMENT AND PROCEDURE Resistivity Measurements

Electrical resistivity measurements were performed using a tailor made device with a Keysight E4980A Precision LCR meter at 1V and 1 KHz. In this device two parallel plates are used as current electrodes, and ΔV measurements are made using several 2mm thick ring electrodes spaced by 3mm electrical insulation. The classical 4 contacts resistivity method presented by Garrouch and Sharma [8] is used.

Saturation Measurements

Saturation measurements were performed using NMR profiling on a 2MHz Geospec from Oxford Instruments, equipped with Green Imaging software. The classical SE-SPI saturation profiling proposed by GIT has been used. NMR profiling allows the measurement of the volume of water in the sample as a function of the position along the sample. For a given sample, an NMR profile is acquired at Sw=100% and after each centrifuge step. Therefore, the saturation profile at any given centrifuge step can be obtained by dividing the NMR profile by the NMR profile at Sw=100%.

PROCEDURE

<u>Sample characterization</u>: 1) Clean with sequences of toluene and iso-propanol; dry by nitrogen flushing then heating at 80°C. 2) Measure permeability and Helium porosity. 3) Saturate with synthetic brine (Sw=100%) to determine pore volume.

<u>Fully saturated properties:</u> 4) Determine brine resistivity Rw. 5) Measure NMR and resistivity profile @ Sw=100%.



plug, then the calculated NMR Sw for each slice, and finally the measured Rt.

<u>Determination of first drainage PCRI curves:</u> 6) Spin the core in the centrifuge in drainage mode until stabilization of the production during few hours. 7) Obtain the NMR and resistivity profile. 8) Loop to step 6 for next centrifuge speed.

<u>Determination of imbibition PCRI curves:</u> 9) Perform a capillary rise experiment putting the core in a foot bath (3mm) monitored with a balance. 10) Measure the NMR and resistivity profile. 11) Spin the core in the centrifuge in imbibition mode. 12) Measure the NMR and resistivity profile. 13) Loop to step 11 for next centrifuge speed.

VALIDATION OF THE TECHNIQUE ON OUTCROP ROCKS

In order to validate the UFPCRI technique, electrical measurements were performed on the same samples, with the continuous injection (CI) method [9]. The classical porous plate was not considered due to the long time required to get the results.

Two outcrop samples (diameter 38mm and length 45mm) were selected: Bentheimer sandstone (28p.u., 2.2D) and Estaillades carbonate (31p.u. & 288mD). All measurements were performed at ambient conditions using mineral oil (non wetting fluid) to displace brine (wetting fluid), using confining stress of 15 bars only for CI.

Once the experiment was completed, the samples were cleaned, dried and saturated to 100% with the same brine used in the continuous injection technique. Then, the UFPCRI method was applied. The experiment was conducted twice in drainage mode, using gas and mineral oil. The purpose was to verify that the same Archie exponent "n" was found for both cases and to show that there was no experimental bias due to the fluid used.

With this technique, one centrifuge step would be enough to determine Archie's saturation exponent "n" (*i.e.* the slope of the RI=f(Sw) log-log curve). This is due to the multiple resistivity measurements on the different slices of the sample. However, it is better to cover a larger range of water saturation to determine "n". For this reason we performed 3 centrifuge steps to investigate a wider Sw range and to derive the capillary pressure curve.

Figure 2 shows the resistivity index vs water saturation plot, for each plug, using data derived from measurements with the CI method and the proposed UFPCRI with oil/brine and gas/brine couples. We observe that:

- The two UFPCRI results (oil/water and gas/water) overlay for both Bentheimer and Estaillades. This confirms the independence of the method versus the fluid used.
- UFPCRI gives consistent results when compared to the CI method for both samples.
- For Bentheimer a deviation from linearity on the CI data is observed. This is a commonly known issue of the CI method [9]. In any case, the determination of "n" being done by a linear regression forcing the zero intercept, the high Sw part of the curve has almost no effect on the "n" value obtained.

Despite the overlay of the experimental data, derived from both methods, the resulting saturation exponent are not identical. Thus, the impact on the calculation of saturation was also verified. This is important for small values of Sw, corresponding to the oil zone in a reservoir.

In order to validate the technique, we need to verify not only the overlay of the experimental data in the RI vs Sw log-log plot, but also the impact of the different values of n found on the saturation calculations, particularly for the small values of Sw, corresponding to the oil zone in a reservoir. For this, Sw calculated using the different values of n are compared, at a mean Sw of 10s.u.:

- For the Bentheimer sample, the maximum difference between the UFPCRI and CI is $\Delta n=0.05$. This translates to $\Delta Sw=0.7$ s.u. at Sw=10s.u.. For higher Sw, the maximum error remains below 1.1s.u..
- For the Estaillades sample, the maximum difference between the UFPCRI and CI is $\Delta n=0.06$. This translates to $\Delta Sw=0.6s.u.$ at Sw=10s.u.. For higher Sw, the maximum error remains below 1.0s.u..



Figure 2: Comparison of Resistivity Index curves obtained by CI method (Oil/Water) in green and our UFPCRI method for both Oil/Water (red) and Gas/Water (blue): Bentheimer (left) and Estaillades (right).

The observed repeatability of the UFPCRI method regardless of the fluid used (gas or oil) and its good agreement with the CI method, clearly validate the UFPCRI technique.

The proposed method allows the joint determination of the Archie parameter "n" and the capillary pressure curve (as presented and already validated by Green [6]) in less than a week for drainage, compared to:

- Several weeks for RI only using the CI method,
- Several months for RI and Pc with the porous plate method.

APPLICATION TO REAL CASES

The UFPCRI method has been applied to a set of three reservoir samples. These rocks are heterogeneous carbonates. As shown in Figure 3, these limestones do not have the same amount and size of vugs. Observing their pictures they can be sorted as sample B the most heterogeneous with the presence of many surface vugs (up to 1mm) while sample A and C seem to be more homogenous with less pronounced vugs. Their basic petrophysical properties are summarized in Table 1.

Drainage (Gas displacing brine saturated sample)

All measurements were performed at ambient conditions. The samples were centrifuged in drainage mode at various speeds (1000, 1200, 1500, 1800, 2500, 3250, 7500rpm). Each time the samples were spun until stabilization of the production. NMR and resistivity profiles were then measured.

Figure 4 presents the drainage results: NMR profiles at various rotating speed are presented in the left column; while the resulting RI curves are presented in the middle column and the resulting Pc curves in the right column.

Because the three samples have different permeability, 7 centrifuge steps were required to cover the whole range of saturations.



Property	А	В	С
Porosity / p.u.	20.5	23	27.2
Permeability / mD	80	12	4
Diameter / mm	38.12	38.11	37.72
Length / mm	40.7	38.53	28.18

Figure 3: Pictures of the samples used. From left to right: sample A, B and C.

Table 1: Basic petrophysical properties of the three reservoir samples



Figure 4: UFPCRI results for drainage of samples A, B and C. NMR profiles (left), resulting RI curves (middle) and Pc curves (right)

The number of NMR profiles is not the same for all the samples. These types of measurements were only performed if significant brine production was observed between the centrifuge steps. Moreover, the NMR acquisition time for samples containing limited amount of water was too long (up to days for a SNR close to 100).

In this case, if there was not enough production for a given sample at a given centrifugal speed, neither saturation profiles nor resistivity profiles were acquired. This explains, for example, why there is no data presented for samples B and C at 7500rpm. These latter did not produce enough brine from 3250rpm to 7500rpm and the NMR acquisition time for samples containing limited amount of water were too long. So samples B and C have also been spun to 7500rpm but no data is presented.

From the NMR profiles, sample A seems homogeneous, while sample B shows heterogeneities and sample C is the most heterogeneous. The impact of the heterogeneity on the results will be discussed below in a separate section.

The RI curves show a straight line for the three samples, verifying the validity of Archie's law for these samples. Sample B data dispersion will be covered in the heterogeneity section. The following Archie's saturation exponents can be derived:

- For sample A: $n_{drainage} = 2.47$
- For sample B: $n_{drainage} = 1.31$
- For sample C: $n_{drainage} = 1.50$

Capillary pressure curves were computed using the Green-Imaging GIT-Cap-Pressure software. The shape of the Pc curve for sample C can be attributed to the heterogeneity and will be discussed in detail later in this paper.

With the centrifuge steps used, many points overlap. Thus this result could have been achieved with a reduced number of centrifuge steps. As proposed by Green *et al.* [6], as few as three centrifuge steps can be used for this purpose.

Imbibition (Brine displacing gas from the sample at residual water saturation)

Once the drainage was completed, the next step was to perform the spontaneous imbibitions by capillary rise to create a saturation profile. Then, NMR and resistivity profiles were measured.

Imbibition under centrifugation was performed at 800, 1000 and 2500 rpm. The criteria to change the centrifuge speed were the same as used during the drainage experiments.

Figure 5 presents the results for imbibition. NMR profiles at various rotating speed are presented in the left column, while the resulting RI curves are presented in the middle column and the resulting Pc curves in the right column.

As expected, the heterogeneities highlighted on the NMR profile during drainage were also observed during imbibition. Sample C is clearly the most heterogeneous of the group. The heterogeneity will be discussed in a dedicated section. As shown in Figure 5, a peak at the top of plugs B and C can be observed. This peak increases with the centrifugal speed despite the fact that at this position Pc is close to 0. This could be attributed to remaining water droplets in surface vugs of the samples which may be blocked during the manipulation between the centrifuge and NMR measurements. The values from the extremities were not considered in the calculation of either the Pc or RI.



Figure 5: UFPCRI results for imbibition of samples A, B and C. NMR profiles (left), RI curves (middle) and Pc curves (right)

RI curves show a straight line for the three samples, verifying the validity of Archie's law for these samples. A greater dispersion of the points in the RI plot can be observed for sample B; this will be discussed in details in the section dedicated to heterogeneity. The following Archie's saturation exponents can be derived:

- For sample A: n_{imbibition} = 1.70, while n_{drainage} = 2.47
- For sample B: $n_{\text{imbibition}} = 0.77$, while $n_{\text{drainage}} = 1.31$
- For sample C: $n_{\text{imbibition}} = 1.09$, while $n_{\text{drainage}} = 1.50$

Pc curves were computed using the GIT-Cap-Pressure software. The surprising shape of the Pc curve for sample C can be explained by heterogeneity. Its small length highlights the limit of UFPCRI to describe a whole range of Sw especially with local heterogeneity.

DISCUSSION

For drainage, the following observations can be made:

Sample A: the most homogenous based on the NMR profiles at 100% Sw, n_{drainage} =2.47. The RI vs. Sw plot shows good linearity. Despite having the largest permeability (80mD),

Swi (37 s.u) is higher than the rest of the samples with lower permeabilities.

Sample B and C: NMR profiles highlight their heterogeneities and identify sample C as the most heterogeneous. Their saturation exponents, respectively $n_{drainage} = 1.31$ and $n_{drainage} = 1.50$, seem relatively low when compared to sample A. Furthermore, their Pc curves reach similar Sw.

For the imbibition curves:

Samples A and C: $n_{imbibition} = 1.70$ and $n_{imbibition} = 1.09$, a significant ratio of approximately 1.4 is observed against drainage, and a poorer linearity than others plugs for sample A. Furthermore, NMR profiles and Pc show that its irreducible gas saturation seems close to zero with $\langle Sw \rangle = 93\%$.

Sample B: $n_{imbibition} = 0.77$, a large ratio of 1.7 is observed between drainage and imbibition. The trend follows a pretty good linearity with a really good match between slices close to zero.

These results emphasize the importance of measuring the saturation exponent for both drainage and imbibition.

All the samples show the same behavior upon the transition to the imbibition cycle: the points, in the RI vs. Sw plots, corresponding to the imbibitions are always located below the ones measured in drainage. This type of hysteresis has already been observed ([10], [11]). Such behavior was successfully modeled by Toumelin *et al.* [12] and Man [13].

Comparison of experimental data with the model available in the literature requires NMR profile at Swi, which was only measurable for Sample A. During measurements, the saturation profile was not acquired at maximum centrifuge speed for Sample B and C as mentioned above. Hence, we will focus the discussion of hysteresis between drainage and imbibition of Sample A. As shown in Figure 6 the RI data for sample A follow the trends modeled by Toumelin[12] for a water wet system. In fact, the drop of n could be described as a water film thickening. Following this theory, the imbibition in Figure 6 could be decomposed into three parts:

- P1: the water film thickness increases rapidly at the pore throat,
- P2: rising of surface layers,
- P3: water film thickening reaches a critical gas saturation where the air phase is disconnected.



Figure 6: RI data acquired on sample A (left) and the model proposed by Toumelin [12] (right).

Heterogeneity:

The UFPCRI method provides directly the local saturation and resistivity. Therefore this method is independent of the Sw heterogeneity.

Heterogeneity of the studied samples can be inferred from the NMR profiles at Sw=100%, and after each centrifuge step. For sample C, the most heterogeneous, NMR profiles shows bumps and valleys; certain regions of the plug de-saturate faster than others despite the Pc gradient imposed by centrifugation.

Figure 7 shows NMR profiles and the resulting Pc curve for sample C where two trends can be observed and each response can be attributed to a particular section of the sample. Considering only the bottom part of the plug (orange rectangle in NMR profiles), all the points in the Pc curve corresponding to this section fall on the orange line. Similarly the Pc curve corresponding to the middle of the plug (red rectangle in NMR profiles) is represented in red. Thus, the shape of the Pc curve for sample C is not due to uncertainties of the technique but rather reveals the heterogeneity of the sample. Additionally, data from both trends could be used to derive Pc curves for both "rock types". The heterogeneity observed in the Pc curve of sample C does not translate into the RI curve as the RI vs. Sw plot showed linearity between the different values. This means that the two "rock types" present in the bottom and the middle of sample C have different Pc curves, but have the same electrical characteristics.



Figure 7: NMR profiles from sample C (left) and the resulting Pc curve. Pc curves obtained from the bottom of the sample (orange rectangle) and the middle (red rectangle) are drawn in corresponding colors.

An interesting result was obtained on sample B for drainage and imbibition: RI curves in Figure 8 were presenting dispersed points (i.e. the points were not aligning clearly on a straight line). However, two trends can be observed when plotted as a function of the position of the measurement. While the Slices 1, 2, 3 & 5 behave linearly in the RI plots; Slices 4 and 6 have a different behavior with flattening of the RI curves at low Sw.

Neither Pc curve nor NMR profiles showed similar deviation for this sample. The non-Archie behavior observed on slices 4 and 6 could be attributed to a dual porosity system in this vuggy carbonate. Such behavior has previously been reported by Fleury *et al.*[14]. Indeed, Fleury proposed a model to fit the results and obtain a non-Archie law [15]. This example demonstrates the capacity of the UFPCRI technique to capture heterogeneities.



Figure 8: RI curves obtained on sample B in drainage (left) and imbibition (right). Slice are sorted from the bottom (slice 1) to the top (slice 6) of the sample.

CONCLUSION

We present for the first time an experimental method (UFPCRI) allowing the simultaneous acquisition of capillary pressure data and resistivity index in a matter of weeks (drainage and imbibitions). This technique combines the method proposed by Green [6] to derive capillary pressure with a novel approach to provide resistivity index. The electrical measurements were clearly validated by comparing the results with the continuous injection method on outcrop samples, and a set of results are presented on heterogeneous carbonates. We present numerous centrifuge steps in order to have an extensive dataset, but three well chosen centrifuge steps could be sufficient to acquire enough data, therefore shortening even more the acquisition time.

UFPCRI has the advantage of avoiding the porous plate and its drawbacks (loss of capillary contact, long equilibrium time, risk of leaks).

Additionally, *classical methods* (porous plate, continuous injection) are completely blind against the heterogeneity of the sample since they report only the *average* values of: capillary pressure, saturation and resistivity. On the other hand, we propose an innovative technique *UFPCRI* which allows the measurement of the *local* properties (porosity, saturation, capillary pressure and resistivity). While core samples are often heterogeneous even at the plug scale, UFPCRI captures local properties and provides reliable petrophysical answers. Would the heterogeneity be a perturbation or a zone of no interest (*e.g.* shale layer), UFPCRI could capture it and discard it from the interpretation.

In summary, the proposed method:

- Is very fast, and therefore can provide Pc and "n" data in the range of operational timeframe, allowing the n results to effectively be used in log interpretation.
- Is able to capture heterogeneity, therefore leaving the choice to the interpreter to use or discard this information in the global n and Pc.

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