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Evaporation Process and Pore Size Distribution in Tight Sandstones: A Study using NMR and MICP

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Abstract

The phenomenon of evaporation is widely observed in a variety of processes and draws attentions from researchers. In the petrophysical study of sandstone, the knowledge of evaporation is necessary to understand the fluids distribution in the rock. Besides, evaporation method was used in the determination of irreducible saturation in lab and removing water blocking in gas well. In this study, Nuclear Magnetic Resonance (NMR) T2 distribution was used to determine the water distribution and migration during evaporation. Mercury Injection Capillary Pressure (MICP) was used to calibrate the T2 distribution produced by NMR. Based on the NMR data and MICP data, the absolute pore radius distribution was obtained and the whole evaporation process of tight sandstones can be divided into three stages. In the tight sandstone samples, the threshold pore radius for the initial evaporation is larger than 0.8 μm .

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Keywords: tight sandstone; evaporation; gas well; pore size distribution.

1. Introduction

The phenomenon of evaporation is widely observed in a variety of processes and drew huge attention from researchers. In the petrophysical study of sandstone, the knowledge of evaporation is needed to understand the fluids distribution in the rock. For example, Messer (1951) and Dodd (1951) introduced the evaporation method to determine irreducible water saturation and proved a good correlation with porous-disk method. Another example would be the Formation Heat Treatment by Jamaluddin (1998) which employed the electrical heater to evaporate the

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trapped water thus increases the gas relative permeability. In terms of irreducible water saturation, the conventional method is to use centrifugal experiment which involves producing free water by the high speed centrifuge running long experimental time. Similarly, the evaporation method was useful in obtaining irreducible water saturation. By recording the water weight retaining in sandstone and time at a certain temperature, the first constant evaporation rate was spotted and the according water amount was the irreducible water saturation. This technique, to some extent, was less studied, but its accuracy is as good as porous media plate and its reproducibility is satisfying (Dodd, 1951). Nuclear Magnetic Resonance (NMR) has been widely used in petrophysics. The magnetic interactions between protons result in longitudinal relaxation (T1) and transverse relaxation (T2). Both T1 and T2 can be used to estimate irreducible water saturation for sandstone or carbonate rock. The study on the behavior of water in the sandstone during evaporation is based on these studies.

In the fully saturated sandstone sample, there is relationship as below: $\Phi = \text{BVI} + \text{FFI}$,

where, Φ is the total porosity, BVI is the Bound Volume Irreducible and FFI is the Free Fluid Index.

2. Evaporation in Porous media

The previous studies on drying process have revealed some processes in evaporation. By some researchers, two stages were divided as a result of evaporation rate: the first stage is a constant rate stage, which is named “surface evaporation”; and the second one is the falling rate period, which is named “internal liquid diffusion”, the boundary between them is named “critical point” (Dodd, 1951; Sherwood and Comings, 1933). The second stage can be divided into another two stages (Shokri et al., 2010) (Figure 1): Stage IIa is the profile-controlled stage; Stage IIb is the slow rate period or vapour diffusion stage.

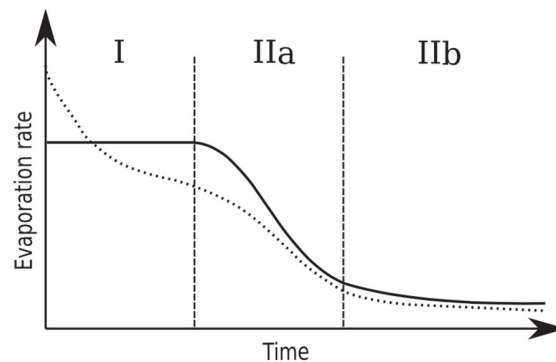


Figure 1 Three stages of evaporation (Shokri et al., 2010)

During Stage I period, the fine pores are hydraulically connected and supply evaporative demand at the surface by drawing water from the inside (by means of migration in the multilayers lining pores of the rock (Shokri and Or, 2011)). The end of stage I is indicated by the disruption of hydraulic continuity in which case the downward gravity and viscous forces are larger than the upward capillary driving force (Lehmann, 2008; Shokri, et al., 2009). Then the evaporation rate will become constant because the diffusion of vapour in the porous media becomes the dominant mechanism after the evaporating menisci retreated deep in the rock (Dodd, 1951).

Messer (1951) conducted the evaporation experiments with different saturants: water, toluene and tetrachlorethane. The characteristic curve of the solid-liquid system was shown as Figure 2. The irreducible volume was determined by using the point of relatively constant desaturation rate.

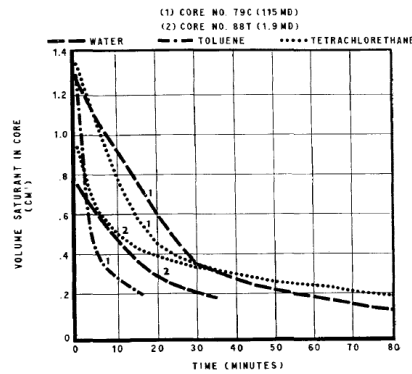


Figure 2 Pore Fluid Retention (Messer, 1951)

3. The Laboratory Measurements

3.1 Experiments Preparation and Setup

The equipment used to obtain the T2 relaxation is a Magritek 2MHz NMR Rock Core Analyzer provided by Department of Petroleum Engineering in Curtin University. The running temperature of NMR was at 30°C. The centrifuge used in this study was a VINCI refrigerated centrifuge. The tight sandstone plug has been saturated with 20,000ppm brine in a saturator pressurized to 2000psi for 24 hours. The properties are listed in Table 1.

Table 1 Dimension and Petrophysical Properties of Tight Sandstone Samples

No.	Bulk Volume(cm3)	Pore Volume(cm3)	Porosity (%)	Permeability(mD)	Weight Dry(g)	Weight Saturated(g)
WR09	73.101	5.843	7.993	0.366	178.6169	185.2243
WR11	56.064	5.444	9.711	0.223	135.3380	142.027
WR12	55.552	1.855	3.34	0.109	141.500	143.598

3.2 Experimental Procedures

The fully saturated sample was taken out from the jar and weighted before conducting the NMR measurements. The NMR rock analyser was set as Table 2:

Table 2 NMR Rock Analyser's Parameters.

Parameter	Inter-experiment. Delay (ms)	Dummy scans	Number of Echoes	Tau (us)	Points per echo	Echo Shift (us)	Dwell Time (us)	Minimum SNR
Value	1000	1	5000	50	12	13	0.5	200

After the first NMR measurement, sample was put into a lab oven, which was preheated to 50 °C. In the first step, for every 5 minutes, sample was taken out to weigh sample mass and conduct NMR test and weigh again, and then sample was put back to the lab oven for drying. This was repeated for 60 minutes. In the second step, the procedures abovementioned were repeated every 15 minutes. This stage last for 120 minutes. The third step repeated the same procedures for every 30 minutes and last for 120 minutes.

4. Experiment Results and Analysis

4.1 Evaporation curve

The evaporation curves for WR-9, WR-11 and WR-12 (Figure 3) were plotted in order to calculate and show the change of evaporation rate and water content in the sample. The water content in the sample is 6.6074g, 6.6890g and 2.098g for WR-09, WR-11 and WR-12 respectively. Based on these curves, the irreducible water saturation can be determined as 47.22%, 49.40% and 44.32% for WR-9, WR-11 and WR-12 respectively using Messer's Method (Messer, 1951).

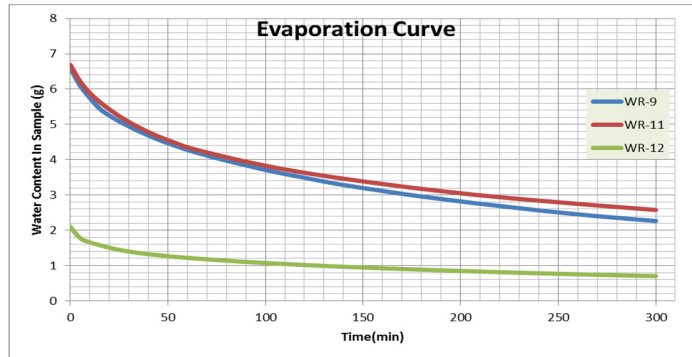


Figure 3 Evaporation Curves.

4.2 NMR T2 Relaxation during Evaporation

During the evaporation experiments, the NMR technique was employed to determine the water saturation in the samples. As the evaporation proceeded, the water in some pores or pore throats migrated inside or outside the pores. By planning suitable NMR tests during evaporation, the behaviours of water migration are possible to be obtained. The measurements showed the details of water distribution during evaporation. Three stages can be divided by using both the evaporation curve and NMR data. The first stage is the evaporation of water in the large and medium-sized pores; the second stage is the evaporation occurs only in medium-sized pores; the third stage is the evaporation in the medium and small-sized pore. From the data in WR-09 incremental spectrum, the heating time during 5~30min can be categorized in the first stage; 35~75min is the second stage; while 90~300min is the third stage. In detail, in the first stage, water in the large pore which has a long T2 relaxation, evaporates causing the shift and reduction of the amplitude of peak larger than 10ms, meanwhile, the amplitude of peak between 1~10ms decrease with time. Although the peak at the shorter T2 changes, it does not constantly decrease but fluctuates. In the second stage the water content in the large pores kept at a low value, the evaporation mainly occurred in the medium-sized pores (around 1 ms). Note that the water content in the small pore stays relatively steady. The discontinuity of water in the sample could occur in this stage. In the third stage, the evaporation in the medium-sized and small-sized pores or pore throats dominates. The peak at around 1.5ms drops with time while the peak at around 0.1ms tends to be disappeared indicating the drying of smaller pore or pore throats. According to the theory of (Lehmann, 2008; Shokri et al., 2009), the start of the third stage shows the discontinuity of water in the sample, so this indicates the end of surface evaporation.

Similarly, for the sample WR-11, three stages can be divided: the first stage is 5~30min; the second stage is 35~90min; the third stage is from 105min to the end of evaporation. In the sample WR-12, the pore size distribution is different from the previous two samples: the relative smaller pore size dominates in the sample. Three stages can be identified: the first stage (0min - 15min); the second stage (20min - 55min); the third stage (60min - 300min).

4.3 Centrifuge Experiments and NMR

These two samples were fully saturated using brine. The T2 distributions, both incremental and cumulative spectrum, were obtained firstly. After that, samples were put into the centrifuge for 24 hours to produce the moveable water from the cores, and then measure the samples T2 distribution. According to (Morriss et al., 1997), the T2 cutoff is determined by the best agreement between the free-fluid porosity and the volume of water produced by centrifuge, so T2 cut-off values can be obtained by plotting these data, and they are 5ms for core WR-09 and 7ms for core WR-11. The irreducible water saturation produced by centrifuge can be reached by evaporation for around for about 30 minutes for WR-09 and WR-11.

The corresponding water saturation is 80.34% and 80.00% respectively. For WR-12, the evaporation method took around 15 minutes to reach the same water saturation (75.9%) as centrifuged sample. For evaporation method, 30 minutes falls on the end of first stage of WR-09 and WR-11, and 15 minutes falls on the end of first stage of WR-12. In this stage, water in the large pore was evaporated which is similar to the phenomenon that water was produced from large pores well connected to the surface during centrifuging samples.

Dodd (1951) took the point when the rate of evaporation first becomes steady to represent the minimum interstitial water content. We use the same way to calculate the point to determine BVI. For WR-09, WR-11 and WR-12, they have the similar results at 160 minutes: the determined irreducible water saturation is 47.22%, 49.40% and 44.32%. This falls into the third stage mentioned in Section 4.2.

4.4 Mercury Injection Capillary Pressure (MICP) tests and NMR tests

NMR T2 distribution can be used to estimate the absolute pore size distribution accompanied with MICP data. When shifting T2 distribution by an effective surface relaxivity, the T2 distribution and MICP data overlay each other (Coates et al., 1999). In this study, we conducted the MICP test for two samples, which locates close to WR-09, WR-11 and WR-12.

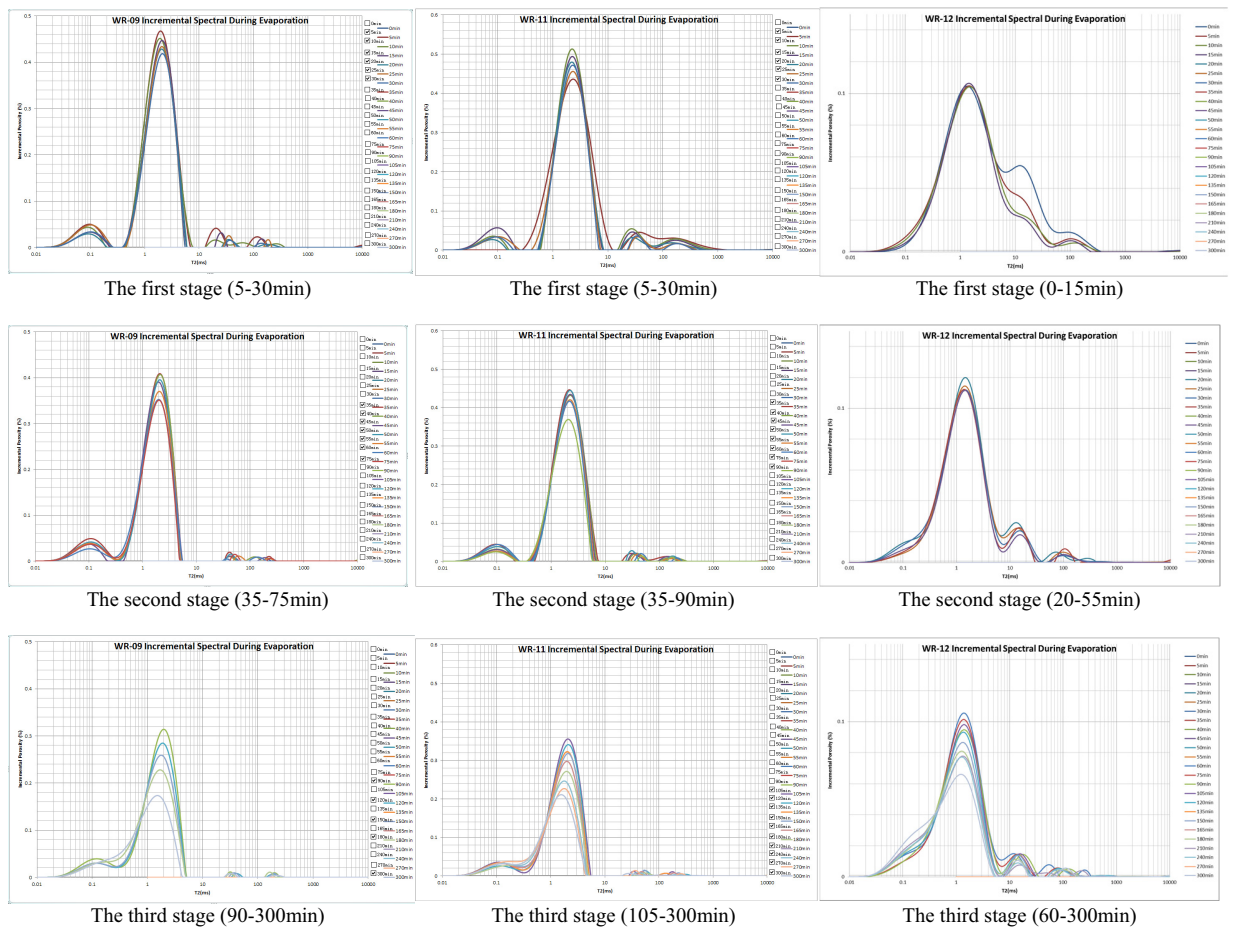


Figure 4 Three Stages during Evaporation in Tight Sandstone Samples.

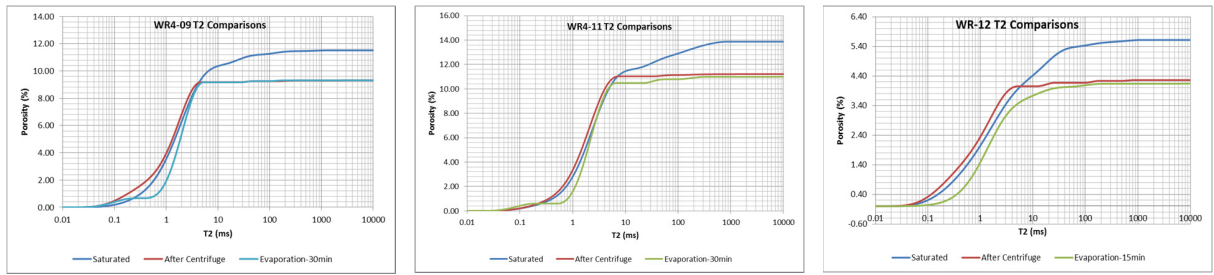


Figure 5 Comparisons of T2 Distributions between Centrifuge Method and Evaporation Method.

NMR T2 distribution and MICP are plotted for each sample for the determination of effective relaxivity (Figure 6). Based on the equation for converting MICP pore radii to T2 value (Marschall et al., 1995): $T_2 = 1000r/2\rho_e$. T2 is the NMR relaxation time, in ms; r is the radius obtained from mercury injection, in μm ; ρ_e is the effective relaxivity in $\mu\text{m/s}$. ρ_e for sample WR-7 is 26.316 and for WR-10 is 28.125 $\mu\text{m/s}$. This study used the average of them 27.2 $\mu\text{m/s}$.

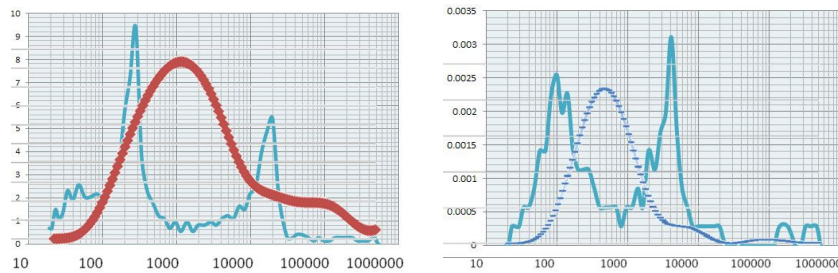


Figure 6 Correlation of MICP and NMR results for Sample WR-7 (Red is NMR) and WR-10 (Blue is NMR).

As discussed in the previous sections, there are three stages during evaporation. When considering the pore radius, in the first stage the evaporation occurred in the pore with radius form 0.816 μm to 16.32 μm ; the second stage from 0.01632 μm to 0.3808 μm ; the third stage from 0.00544 μm to 0.3808 μm (Figure 7). The threshold pore radius for the initial evaporation is larger than 0.8 micron in tight sandstones.

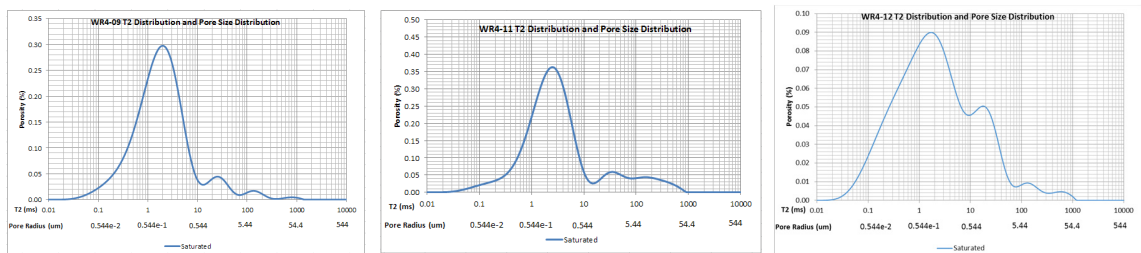


Figure 7 T2 Distributions and Pore Size Distribution.

5. Conclusions and Discussions

The evaporation method can be an effective and efficient method in petrophysical study of reservoir rocks. Based on the preliminary study on the evaporation process and pore size distributions of tight sandstones, we come to the following conclusions:

1. The evaporation process of tight sandstone can be divided into three stages according to the water evaporation behaviour in pores of different sizes.

2. By heating tight sandstone samples to the end of first stage (for WR-09 and WR-11, it was about 30 minutes while for WR-12 it was about 15 minutes), the water saturation reached to a value (80.34% and 80.00% for WR-9 and WR-11, 75.9% for WR-12) equivalent to that prepared by a 4500rpm centrifuge.
3. By using Messer's Method, the irreducible water saturation determined by evaporation is 47.22%, 49.40% and 44.32% for WR-9, WR-11 and WR-12 respectively.
4. The first stage evaporation occurs in the pore with pore size larger than 0.8 micron.

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