IN SITU SATURATION MONITORING (ISSM) – RECOMMENDATIONS FOR IMPROVED PROCESSING

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ABSTRACT

In situ saturation monitoring (ISSM), using X-rays or gamma rays, has become a common method to determine fluid saturations in commercial coreflood experiments. The most common method in commercial laboratories entails 1D saturation measurements as a function of core plug length and of experimental time. Laboratories often employ ISSM as the only method of determining fluid saturations, assuming an almost infallible accuracy of 1-2 saturation units (s.u.). However, as for all measurement methods, there are possible sources of uncertainty in ISSM data. Previous papers have discussed some of these uncertainties, such as X-ray drift (Coles, et al. 1995), and inappropriate calibration scans or changes to core or fluid properties during testing (Cense, et al., 2014). Despite this evidence, some laboratories continue to use ISSM measurements alone, assuming negligible uncertainty.

In the authors' experience, uncertainties not only exist in measurement errors, but also may be introduced by inappropriate processing and interpretation methods. This paper first considers the stipulated 1-2 s.u. accuracy and the necessary signal-to-noise ratio, i.e. counts required, to achieve this; as well as providing a suggested approach, where plausible, to correct saturation data compromised by incorrect calibration scans. It also considers the uncertainties in use of ISSM production volumes in determining unsteady state relative permeability; specifically, pre- and post-breakthrough data and the assumptions surrounding selection of breakthrough from flood front scans. In addition, ISSM profiles are often used in coreflood simulation of relative permeability to aid correlation of the capillary end effect; incorrect data processing may compromise this correlation. In conclusion, the paper considers several sources of error in ISSM data and provides a recommended approach to acquisition, processing and interpretation of ISSM data for calculation of fluid saturations.

INTRODUCTION

In situ saturation monitoring (ISSM) was introduced to the oil and gas industry in 1946 by Boyer, et al.¹, with suggested improvements by Morgan, et al.² in 1950 and supported by further experimental data in Geffen and Gladfelter³. Since this time, in general, laboratory equipment has developed and improved to reduce some of the potential uncertainties: radiation sources and detectors are more stable (possibly due to more stable power sources), core holders have been developed using lower attenuation materials

and/or with thinner walls to reduce background noise, temperature regulated equipment reduces large fluctuation in attenuation due to temperature, etc. However, despite improvements, there remains potential for uncertainty in attenuation measurements from various factors, including: temperature variance, radiation source age and/or degradation, power fluctuations, core plug heterogeneity, core plug location displacement and random noise. Coles, et al.⁴ recommend some techniques to improve accuracy for X-ray systems: scan a fixed reference material immediately prior each core scan and use the value to calibrate each scan, use slow warm-up times for X-ray tubes to improve source stability and extend their life. Use of a reference material scan enables corrections also for gamma-ray systems - for temperature drift and source degradation. Cense, et al.⁵, in addition to temperature variance, noted other potential uncertainties: changes to the rock matrix during testing, errors in calibration scans (i.e. the scans performed at 100%) saturation of the individual fluid phases), limited attenuation difference between the fluid phases, component transfer between fluid phases, and percentage of the core diameter captured within the X-ray (or γ -ray) stream. The paper recommends that, in addition to recommendations by Coles, et al., ISSM data should be supported by an alternative method of determining intermediate (i.e. during coreflooding) saturations, as well as verification of endpoint saturation(s) by an alternative method. Despite evidence to the contrary, some laboratories continue to assume negligible uncertainty for ISSM data and employ only ISSM saturation, without alternative verification.

In the authors' experience, ISSM saturation uncertainties are not only introduced from mechanical, experimental, physical and/or chemical variances, but may also be introduced through inappropriate data processing and interpretation methods. Few literature articles have considered these uncertainties. First and foremost, it is essential that sufficient X-ray (or γ -ray) counts are collected at each location to ensure that the measurement variance is within a predetermined percentage of the attenuation contrast between the fluids (i.e. usually 1-2%), this may not always be the case. The method of determining which scan locations are extraneous (i.e. which locations are not measuring core plug data) may lead to errors in average saturation calculations and may introduce uncertainty into the process of correlating capillary pressure and relative permeability in coreflood simulations.

GENERAL ACCURACY CONSIDERATIONS

The use of ISSM techniques to determine the saturation profile in core flooding experiments should always be considered if budget allows. It is the only method to confirm that a core sample behaves as an homogeneous rock and to observe the capillary end effect. If a core sample consists of multiple rock types, different saturations will prevail during a core flood, and different areas in the sample may have different relative permeability. If one assumes that the sample is homogeneous in the interpretation, varying rock types will be ignored, and the result will be an upscaled relative permeability and/or residual oil value, which may be valid for the core sample but not for the rock types.

To allow for good *in situ* saturation measurements, one needs an experiment where only the saturation changes, and other experimental conditions remain stable. This means that:

- 1. the core sample does not dissolve in the fluids used and there is no precipitation: even if minor amounts of minerals leach out or precipitate, this has a huge impact on the saturation calculation due to the much higher X-ray/gamma-ray absorption of rock minerals compared to fluids,
- 2. the core sample should remain in the same place. Some laboratories remove samples from the set-up to clean them. Placing these samples back to their original position is extremely difficult. Even a slight mismatch in position may result in a different rock volume being acquired in the X-ray/gamma-ray beam. This has a huge impact on the saturation calculation for the same reason mentioned in 1,
- 3. the calibration scans of the core fully flooded with oil (or brine) should be done on core samples that are 100% filled with oil (or brine). Often, it is not easy to confirm whether all residual fluids have been cleaned out before flooding the samples with the calibration fluid. A sign that this was indeed a problem is that saturations will be below 0 or above 1. A mitigation will be to repeat the cleaning cycle and remeasure the calibration scans,
- 4. the temperature of the detector must be constant. As X-ray detectors are extremely sensitive to changes in temperature, the detector should be at a constant temperature during the experiment. Since this is a non-trivial task, one can use reference scans to compensate for temperature fluctuations in the room,
- 5. the intensity of the source must be constant. If the intensity varies over the duration of the experiment, the calibration scans will not reflect representative conditions. Again, this issue can be mitigated using a reference scan to compensate for source intensity fluctuations.

In the latter case, the standard equation to calculate saturation from counts (Eq.1) is revised (Eq.2).

$$Sw = \frac{\ln\left(\frac{I_{S_{W}}}{I_{S_{W}=0}}\right)}{\ln\left(\frac{I_{S_{W}=1}}{I_{S_{W}=0}}\right)} \quad (Eq.1) \qquad \qquad Sw = \frac{\ln\left(\frac{I_{S_{W}}}{I_{S_{W}}}\right) - \ln\left(\frac{I_{S_{W}=0}}{I_{S_{W}=0}}\right)}{\ln\left(\frac{I_{S_{W}=1}}{I_{S_{W}=1}}\right) - \ln\left(\frac{I_{S_{W}=0}}{I_{S_{W}=0}}\right)} \quad (Eq.2)$$

Where I_{Sw} refers to the number of counts measured at the detector at a saturation S_w . $S_w=0$ refers to the calibration scan with the core fully filled with oil/gas, $S_w=1$ refers to the calibration scan with the core fully filled with water. The *ref* superscript refers to the reference scan, which can be made on a separate detector. The reference scan is made at the same time as the measurement of the absorption of the core holder (see Figure 1).

Scanning times need to be sufficiently long to allow for precise saturation interpretation. But what is long enough? In an ISSM experiment using an X-ray source, stable saturation at the end of a steady state fractional flow step usually is determined from the average of all slices along the sample length. If the scanning time is too short, the error in saturation may be larger than desired. In the example shown in Figure 2, the error at slice 26 mm initially was 3.5%, and it took about 6 hours to acquire statistically sufficient scans before the saturation error was reduced to less than 2% at individual slices, whilst scanning the whole core plug. This applies to all slices in the core and thus, it can be shown that the error in the average saturation of the core is of the same magnitude.



Figure 1: Schematic top view of the X-ray source, the core sample, the detector and the reference detector.

It requires no complex calculation to check whether the error in saturation is reduced to an acceptable level: simply plot saturation from individual slices (in practice this will be counts, as one usually does not have the calibration scans available at this experimental stage) and calculate the standard deviation. With some experience, the calibration levels of an oil-filled and water-filled core, thus, error in saturation can be estimated. Especially in cases where residual oil/gas is determined, one needs to wait sufficiently long to attain higher accuracy. Merely flooding 1.5 pore volumes in a couple of hours is not good enough.



Figure 2: Saturation (blue), average saturation (green) and standard deviation (red) at a single slice at the end of a steady state step. The initial error in saturation is 3.5%, but it is halved to 1.7% after measuring the saturation for about 6 hours.

SAMPLE LENGTH DETERMINATION

Core plugs are commonly loaded into cylindrical rubber sleeves between two endstems usually composed of high grade steel or corrosion resistant alloy, with high attenuation coefficient, see Figure 3(A). An X-ray (or γ -ray) source is situated on one side of the core with a detector linked directly opposite and both attached to the same mobile unit on a motorised track. The unit can move backwards and forwards, or up and down, depending on equipment orientation, but the source and detector are always in the same relative position, i.e. detector directly opposite source. During data acquisition, either a voltage is applied (X-ray) or a slot or pin-hole opened (γ -ray) to allow radiation to be emitted from the source towards the detector. Radiation reaches the detector through a slot, which most often transcribes as a 2 mm slice. The source/detector unit is then moved 2 mm and counts recorded from that next 2 mm slice. As indicated in Figure 3(B), the number of counts accumulated through the endstems is lower than those accumulated through the core plug. There is interference from the endstems into the measurements at the boundary between endstem and core plug. The interferenge causes some potential uncertainty regarding where the sample begins and ends. This uncertainty leads to loss of data at the core plug boundaries.



Figure 3: (A) Example image of core plug loaded inside ISSM coreholder (B) Example X-ray counts at scan locations across the system length.

The approach used by many laboratories to determine the plug boundaries is indicated in Figure 4(A) through (C). The total (or base) count measurements are acquired, and plug ends are determined by using either the initial scan (A) only or all scans (B), as the point of inflexion from high counts towards lower counts, as indicated - locations 7 and 36 along the sample length. Data from extraneous locations (1-6 and 37-44) will not be carried forward into calculations of saturation. Only the data between these inflexion points (7-36) will be used to determine saturation, without reviewing the full dataset. After calculating saturation, any unusual variance near the selected ends may result in additional attrition, e.g. the first location in Figure 4(C), length = 0.248 cm may be negated.

Some labs will assume that the full length of the sample has been acquired, stretching the data to match caliper length. In this process, caliper length will be divided by the number of scanned locations, to produce a scan interval length. Scan location numbers are renumbered from 1 (hence, 7 - 36 becomes 1 - 30). In the example shown, Figure 4(C), caliper length = 7.43 cm, sample scan locations = 30, producing a scan interval of 0.248 cm. The renumbered locations are multiplied by interval length to produce the sample length (0.248 - 7.43 cm). As seen, there is no data between 0 - 0.248 cm, but data incorrectly exists to the full length of the core plug.



Figure 4 (A) Example of base counts, scan locations 1-44 (B) Lab selects sample ends from base counts: locations 7-36 (C) Lab reported saturation data: locations 7-36 stretched to caliper length (D) Saturation calculated from all locations indicating alternative end selection: locations 8-41 (E) Selection of sample ends from D (F) Improved saturation data, actual sample length - not forced to caliper length.

A suggested improvement to this common approach, is first to calculate saturations employing all location data and plot as a function of location, limiting saturation (y-axis) between 0 and 1, as shown in Figure 4(D). This exhibits locations at the extremities to

have nonsensical, non-physical data, below 0 or above 1, obviously invalid. It provides a more appropriate method to determine sample end faces (here shown to be locations 8 and 41) where saturation becomes relatively constant and homogeneous. This approach can often extend the number of selected locations (usually by 1 or 2 locations, i.e. 2 - 4mm), but sometimes by several locations, as in the case shown in Figure 4(E), where 4 scan locations (0.8 cm) were added. Figure 4(F) provides the final reinterpretation of Sw as a function of length. Compared to the lab interpretation, there is a missing interval at the inlet end (close to zero), due to the decrease in Sw between locations 7-8, (approaching 10 s.u. decrease) and deemed impacted by endstem interference. This interval was not removed by the lab and can be observed in Figure 4(C). These different approaches can produce significant variance in calculated saturation. In the presented example, there was an average difference of 3 saturation units (s.u.) and maximum of 6 s.u.

As previously stated, saturation data is not expected to extend the full sample length due to endstem interference. The practice of stretching saturation data to the full caliper length also impacts the observed capillary end effect. In fact, the missing few millimetres at the production end are those most heavily influenced by capillary effects. We would not recommend this practice. We recommend that length calculation be determined by using the standard slice interval of the equipment (usually 2 mm). Most often approximately 2-3 mm may be lost from each end. In the example core plug (Figure 5), the difference between saturation at the endface was observed as Sw = 0.62 (lab approach) and Sw = 0.4, suggested approach.



Figure 5: The production endface showing the different saturation profiles derived from different data processing approaches. At Pc = 0, common lab approach Sw = 0.62, recommended approach extrapolated Sw = 0.4, approx.

WATER BREAKTHROUGH SELECTION

Many laboratories employing ISSM during unsteady state (USS) relative permeability corefloods, use only ISSM as the basis of determining saturation change. Figure 6(A) provides a typical example of saturation profiles acquired during such a flood. The plot shows the original Swi state (blue line) and the progressing flood front (light-grey lines) at increasing time steps. Average Sw from these profiles are often used towards JBN style calculations of relative permeability, but incorporate error since each slice has been

acquired at a different time point not the time used for determining average Sw. The magnitude of this error varies depending upon aspects such as; flowrate, scan number, scan time, front stability, etc. Appropriate time corrections and/or numerical simulations are recommended to synchronise these data before use in interpreting relative permeability. Figure 6(A) also shows the profile most often selected as the point of water breakthrough; the profile when Sw begins to change at the production face (indicated by the dark, dashed line, "Lab selected BT"), the thought being that since water has begun to change at this face, breakthrough must have occurred and both oil and water will now be produced. However, there are two main errors in this approach: firstly, there is unobserved data in the last few millimetres and the final location data may not correspond to the actual production face; secondly, this selection does not account for capillary effects, and a potential that water saturation may first build at the production face prior to actual breakthrough. This case is indicated by the dark-grey, continuous line ("Actual BT") and may correspond to a water-wet case.



Figure 6: (A) ISSM profiles showing typical BT selection versus actual BT profile (B) Example showing the ISSM profiles at water breakthrough, for a range of wetting states, water wet through oil wet. (C) Relative permeability input to A (D) Capillary pressure input to A

The point of water breakthrough depends on variables such as mobility ratio, stability of flood front, heterogeneity, wettability, etc. Assuming appropriate sample selection and quality control, the major variables determining breakthrough in a coreflood will be mobility ratio (viscous forces) and wettability (capillary forces). At the production face, viscous displacement forces approach zero and are less able to overcome non-water-wet

capillary entry pressures. Thus, production face saturation will be strongly influenced by wettability close to Pc=0.

Figure 6(B) shows the variance in possible saturation profiles at the point of breakthrough for different wetting conditions (wetting conditions were represented using a variety of relative permeability, Figure 6(C), and capillary pressure, Figure 6(D), inputs). For the water wet case, as the flood front approached the production boundary end, and prior to water breakthrough, water saturation built, in conjunction with increasing differential pressure, until spontaneous imbibition saturation was achieved. For the oil wet case, water breakthrough was almost immediate upon water reaching the production end, since there were minimal or no spontaneous water imbibition forces.



Figure 7: (A) Unsteady state coreflood production data indicating different breakthrough times, ISSM derived and actual (B) Analytical relative permeability curves (JBN or Jones-Roszelle) derived from the different BT times

In many water-oil imbibition corefloods the experimental time for volumetric oil production (Vo) and differential pressure (dP) may be offset due to time to displace dead volumes and outlet flow lines: inlet dead volume for dP; inlet + outlet dead volume and separator flow line volume (or piping to other volumetric measurement vessel) for Vo. Although there are various methods to align Vo and dP to a mutual timeline, a common method is merely to align the breakthrough point, most often by correcting dP-time to align with Vo-time using the dP point of inflexion, usually maximum dP, as the dPbreakthrough point. However, if Vo-breakthrough has been selected incorrectly, as described above and shown in Figure 6(A), disparity is created. Figure 7(A) shows an example case, where breakthrough was chosen incorrectly from ISSM data (the first scan to exhibit Sw increase at production end) and dP time was offset to match this breakthrough time. Subsequent, standard analytical methods to calculate relative permeability (JBN⁶ or Jones-Roszelle⁷), using this post-breakthrough, time-correlated data will calculate incorrect relative permeability data, as depicted in Figure 7(B). This occurs because a portion of pre-breakthrough oil production is included into the interpretation, producing erroneous data at unusually low saturation values. Although the difference in adjusted time does not appear significant, it has a significant impact on the interpreted data, particularly saturation.

A recommended approach is to correct Vo and dP timelines independently, and quality check the point of breakthrough, preferably using an alternative method to ISSM data:

- 1. Vo-time
 - a. it is essential to know the actual flowrate (either by using volumetrically calibrated pumps, or by determining flowrate from the initial linear production data, where the gradient Vo/time = actual flowrate (Q_{act})
 - b. it is essential to have measured the dead volumes of the system (i.e. production fluid held within flowlines that cannot be bypassed/displaced prior to test initiation)
 - c. Vo-time must then be corrected for dead volumes and regression performed to find the time intercept at Vo=0.
- 2. **dP-time** from knowledge of the inlet dead volume and actual flowrate, dP-time offset is given by $t_{dP} = t_{meas} DV_{in}/Q_{act}$, where t_{dP} is the corrected dP-time, t_{meas} is measured test time (from commencement of flow), DV_{in} is the inlet dead volume and Q_{act} is the actual flowrate, as determined during Vo-time offsets.
- 3. Verify that Vo and dP breakthrough is consistent

ISSM SATURATION CORRECTION

McPhee, et al.⁸ recommends that saturation uncertainty should be within ± 3 s.u., and that saturation should be verified by at least one additional measurement technique. ISSM saturation verification was strongly recommended in Cense, at al.⁵, and we would reiterate the need to corroborate fluid saturation after coreflooding, since ISSM saturation data are not infallible.

If appropriate sample selection has been performed, best practice experimental procedures followed, and quality controls implemented throughout testing, there is generally excellent correlation between ISSM and other methods of determining saturation; particularly at experimental endpoints (test initiation and completion). There may be small differences in intermediary saturations during the steady state method because of system dead volumes and subsequent small errors introduced by the assumptions about how fluids segregate throughout pipework and valves at specific fractional flow rates. Thus, in correctly controlled steady state corefloods, the ISSM saturation data from the intermediary fractional flow rates may be considered as more accurate than the intermediary volumetric data. It is therefore essential to verify the endpoint saturations to validate these intermediary data. The recommended approach to verify saturation, is to ensure that there is full control of saturation data throughout the preparatory stages ahead of the coreflood: saturating the core with formation water and establishing initial water saturation conditions, and throughout the coreflood itself, by verifying final saturation; most often measuring final water saturation (Swf).

From initial knowledge of the sample pore volume, checks should be in place to ensure the sample is fully filled with formation water during the saturation process. Initial water saturation (S_{wi}) must be verified, either by gravimetric or volumetric methods (or preferably both). There are a variety of methods of establishing S_{wi} , but the preferred

method would be an individual porous plate with net confining stress, that allows production volumes to be measured directly into a graduated vessel (usually a glass burette/pipette). Sample weights should be measured before (fully water saturated) and after (at S_{wi}), if possible, to compare S_{wi} volumetrically and gravimetrically.

$$S_{wi} = 1 - \frac{V_w}{V_p}$$
 (volumetric) $S_{wi} = 1 - \frac{(W_{Sw=1} - W_{Swi})}{V_p \ \Delta \rho}$ (gravimetric)

Where V_p is pore volume [cc], V_w is the produced water volume [cc], $W_{Sw=1}$ is fully saturated weight [g] (with formation water), W_{Swi} is the weight at Swi [g] and $\Delta \rho$ is the density difference of the fluid pair [g/cc]. These data should be compared against ISSM derived average Swi data (± 3 s.u.).

After the coreflood, the final ISSM water saturation should be confirmed by an additional measurement technique: volumetric production data, Karl Fischer titration, miscible dispersion analysis. Dean & Stark extraction, etc. Production volumes captured into a graduated and calibrated separator are often an excellent additional measurement and may be used, not only to verify final saturation, but intermediary saturations also; although, volumetric error may be introduced by small system leaks, mass transfer (if fluids are not fully immiscible), droplet retention in the pipework, etc. Karl Fischer titration is an excellent method of determining small water volumes, but may be susceptible to error from; incomplete extraction of the water volume during the solvent injection stage of the process, inadequate mixing and sampling of the solvent/hydrocarbon/water effluent blend, chemical interference if sodium iodide is used. etc. Miscible dispersion could be used to determine the saturation of the mobile fluid phase, but could incorporate significant error dependent on the heterogeneity of the sample and/or fluid flow-path. Dean & Stark extraction is usually an ill-advised method of confirming water volume from ISSM analyses, because the core plug must be removed from the equipment, and replaced exactly to the same location and orientation, which is difficult to accomplish. The impact of removal and replacement is dependent upon the accuracy to which this can be accomplished, the attenuation differences between the different components (core holder, sleeve, core, fluids, etc.) and core plug heterogeneity.

Once all these data are collated, it should be considered which of the data are the more accurate at initial and final conditions, for instance S_{wi} and S_{wf} , respectively. If ISSM data is observed to exhibit significant variance from other verifiable data, the source of the error should be scrutinised, considering the potential sources of error for ISSM data, as previously outlined. However, the endpoint saturations deemed to be accurate might be used to correct ISSM data to determine saturation profiles that can be useful, at least qualitatively. First, calculate normalised water saturation (Swn) based on the ISSM endpoints, then denormalise based on the true endpoints:

$$S_{wn} = \frac{S_w - S_{wi}^{ISSM}}{1 - S_{wi}^{ISSM} - S_{or}^{ISSM}}$$
(Eq.3)

$$S_{w_corr} = S_{wi}^t + S_{wn} \left(1 - S_{wi}^t - S_{or}^t\right)$$
(Eq.4)

Where S_{wi}^{ISSM} is the ISSM based Swi, S_{or}^{ISSM} is the ISSM based residual oil saturation, $S_{w_{-}corr}$ is corrected saturation, S_{wi}^{t} is the true Swi and S_{or}^{t} is the true residual oil saturation.

CONCLUSIONS

In situ saturation monitoring (ISSM) by attenuation of X-rays or γ -rays, may not always provide accurate saturation data. The following recommendations are suggested:

- Optimise readings by including reference scans, to correct for variance in test conditions (hence attenuation variance).
- Obtain several measurements at steady state conditions (at least five), calculate standard deviation and, if necessary, continue measuring until SD is below 0.02 before continuing to the next stage of testing.
- Calculate saturation from all location data (including non-sample data) and use saturation versus scan location to determine inlet and outlet endfaces.
- Do not stretch data to the measured caliper length, but merely use the equipment slice interval (usually 2mm) to calculate from scan number to plug length
- Do not use ISSM to determine water breakthrough; an alternative method should be employed
- Synchronise production and pressure times by using measurements of system volumes and accurate flow rates to determine offsets
- Verify the production and pressure timelines to ensure corroboration at water breakthrough
- Verify saturation at the test endpoints by alternative methods (e.g. volumetric or gravimetric data, Karl Fischer titration, miscible dispersion, Dean-Stark extraction)

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