

HOW RELIABLE IS IN SITU SATURATION MONITORING (ISSM) USING X-RAY?

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ABSTRACT

In core flooding studies, where fluids are injected to mobilize hydrocarbons, X-ray measurements are often used to monitor the hydrocarbon saturations in the core. This is done as a function of the position in the core and as a function of time. The goal is to understand how effective the injected fluids can displace the hydrocarbons, and to measure how much hydrocarbons are left behind in the core. The calculation of in place saturations from X-ray, however, is not straightforward, and it is often unclear what the uncertainties in the calculated saturations are. In this paper, we compare calculated saturations from X-ray with saturations from direct measurement of produced hydrocarbon volumes from the core. The direct measurement is obtained using a novel apparatus which measures the production of oil using a balance under a back pressure. We show that differences between the two methods can be quite substantial and we discuss what may be the reasons causing these differences. The paper ends with a discussion on how we can improve the use of in situ saturation monitoring for unsteady state experiments.

INTRODUCTION

Core samples are often used in experiments with the purpose of determining the residual oil saturation and relative permeability. This information is used in reservoir simulators for example to predict the production of oil/gas in time in order to determine the economic viability of the project. Quantification of the fluid saturations in the core and of the uncertainty therein is therefore important. In most cases where saturations are measured using X-ray, it is unknown what the accuracy of the saturation is (see *e.g.* Trewin, 1992; Maloney, 2002).

Saturation of the core samples is not directly obtained but inferred from the attenuation of X-ray or gamma-rays when they pass through the core holder, the core plug and the fluids therein. The attenuation of the X-ray beams is defined as

$$\frac{I}{I_0} = e^{-\mu d} \quad (1)$$

where I_0 is the intensity of the original X-ray source, I the intensity measured at the detector, μ the attenuation coefficient of the material between source and detector and d refers to the material's thickness. As there is more than one component between the source and the detector, the equation can be expanded

$$\ln\left(\frac{I}{I_0}\right) = -(\mu_{core}d_{core} + \mu_{holder}d_{holder} + \mu_{brine}d_{brine} + \mu_{oil}d_{oil}) \quad (2)$$

where we took the logarithm on both sides of the equation. Each term indicates the contribution of a material (steel, core, fluids) to the attenuation of the X-ray beam. The contribution of the brine to the attenuation can be rewritten as $\mu_{brine} d_{core} \varphi_{core} S_w$. Likewise, the oil contribution is equal to $\mu_{oil} d_{core} \varphi_{core} S_o$ which can be rewritten as $\mu_{oil} d_{core} \varphi_{core} (1-S_w)$. Re-arranging terms gives us the saturation:

$$S_w = \frac{\left[\ln\left(\frac{I}{I_0}\right) + \mu_{core}d_{core} + \mu_{holder}d_{holder} + \mu_{oil}d_{core}\varphi_{core} \right]}{(\mu_{oil} - \mu_{brine})d_{core}\varphi_{core}} \quad (3)$$

In theory, this should work well to determine the water saturation in the core once the attenuation coefficients are known. Attenuation coefficients change with the energy of the X-ray beam. As a normal X-ray source has a distribution of energies, Eq. (3) must be integrated over all energies. In practice, it does not work well, as the attenuation factors have a high degree of uncertainty. Since the coefficients of the core and core holder are so much bigger than that of oil and water, the uncertainty therein disturbs the precision of the saturation calculation to a great extent. For a Hassler type of core holder with a carbon fiber mantle and a 1.5 inch diameter sample the typical values for $\mu*d$ at 65 keV are given in Table 1.

Table 1 Typical values for the linear attenuation of components between X-ray source and detector for a core holder.

	$\mu * d$
core	0.70
holder	1.51
brine (@ $S_w=1$)	0.23
oil (@ $S_w=0$)	0.17
Total	2.61
contrast brine-oil	0.06

To circumvent this problem, the standard practice is to make calibration scans. Calibration scans are made when the sample is completely filled with brine and when the sample is completely filled with oil. During the experiment itself, the attenuation is recorded, which comprises of attenuation of all materials that are in between the source

and the detector. A variation in fluid saturation in the core will lead to a change in attenuation, if the attenuation coefficients of brine and crude oil are significantly different. When the experiment is finished, fluids are removed from the core by flooding with solvents, the core is first saturated with 100% brine and a calibration scan is made. Subsequently, brine is removed from the core by flooding with solvents, the core is fully saturated with oil and another calibration scan is made. Eq. (3) can be approximated by a Taylor expansion (since $|I/I_0| < 1$), and it can then be shown that the actual water saturation in the core at any time during the experiment can be approximated in that case by the difference between the measured intensity at that saturation $I(S_w)$ minus the oil scan $I(S_w=0)$, normalized with the difference between the brine scan $I(S_w=1)$ and oil scan $I(S_w=0)$:

$$S_w = \frac{I(S_w) - I(S_w = 0)}{I(S_w = 1) - I(S_w = 0)}, \quad (4)$$

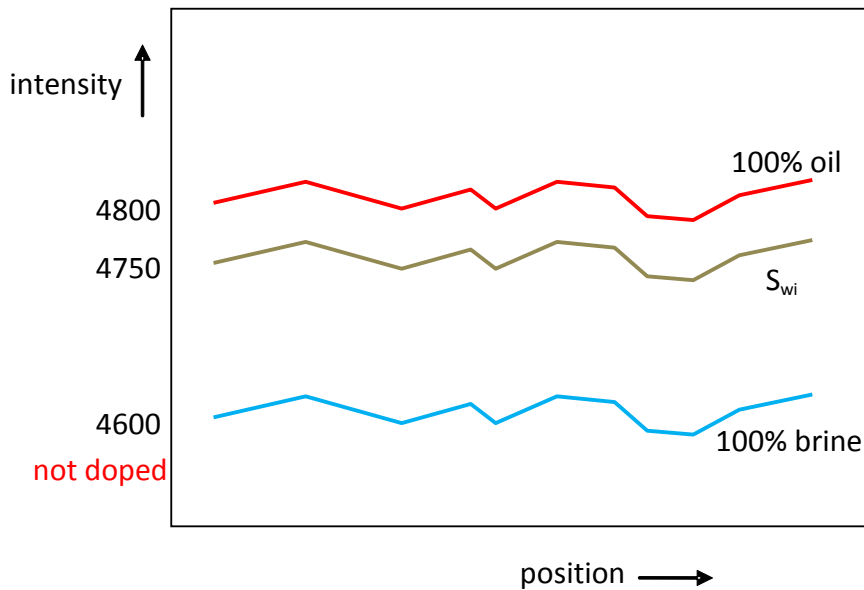


Figure 1 Schematic graph of calibration procedure. Samples are scanned at 100% brine and 100% crude oil saturation. Intermediate saturations can be inferred from (linear) interpolation between the two signals. The initial water saturation profile would correspond to approximately 25% water saturation in this example.

where I represents the intensities measured at the detector for various fluid saturations. By taking long enough (5 min) scans at each position along the core, the statistical error can be reduced to less than 1% water saturation. This can be checked by propagating the errors of equation (4), where x denotes each saturation measurement in Eq. (4):

$$\Delta S_w = \sum_x \left| \frac{\partial S_w}{\partial I_x} \right| \Delta I_x, \quad (5)$$

$$\Delta S_w = \frac{2\Delta I}{I(S_w=1) - I(S_w=0)} + \frac{2\Delta I [I(S_w) - I(S_w=0)]}{[I(S_w=1) - I(S_w=0)]^2}, \quad (6)$$

In our set-up, we take 20 intensity measurements at each scan position, where one measurement is the integrated signal over 166 ms; the standard deviation observed is 30 counts. For our calibration scans we repeat this sequence 80 times, so that we have 1600 measurements at each position. The standard deviation of the mean however, is then equal to

$$\sigma_{mean} = \Delta I = \frac{\sigma}{\sqrt{N}}, \quad (7)$$

where σ is equal to 30 and N is equal to 1600. Using these numbers in Eq. (7) gives a standard deviation of the mean equal to 0.7. Plugging in the numbers from Figure 1 in Eq. (6), using $\Delta I=0.7$ gives an error in the initial water saturation of 0.0088, or 0.9%. It appears that the method of calculating water saturation from X-ray scans is very precise! A similar number for the error in saturation was found by Sharma *et al.* (1997). As one can see from Eq. (6), the error can be reduced even further by increasing the contrast between the oil and the brine. This can be achieved by doping the brine using for example Iodide or by doping the crude oil using for example Iodo-decane (Trewin, 1992). The downside of using these chemicals is that this may influence the wettability state of the rock (Kaliyugarasan, 2013).

Common issues

The use of X-ray to determine the *in situ* saturation of reservoir rocks comes with common issues that may result in larger uncertainties than 1% s.u. (saturation units) as stated before. This may be due to the following:

- changes in rock matrix: if the rock changes during the experiment, it will be interpreted as a change in water saturation. For example, dissolution during the experiments will lead to *higher* counts at the detector, and will be interpreted as (see Figure 1) a *higher* water saturation than the rock has in reality. On the other hand, precipitation of minerals will lead to lower counts, and hence will be interpreted as a *lower* water saturation.
- the calibration scans are made on reservoir rock that does not contain 100% brine (or 100% crude oil). This may result in a systematic error that either overestimates or underestimates the water saturation. We measured the water saturation after a 100% oil scan using Dean-Stark analysis, and observed that for two samples the water saturation was still 0.3% and 2.7%. This caused the oil calibration scan to be too low (see Figure 1), and thus a bias towards *too low* water saturation. To ensure proper calibration, the samples must be cleaned well while they are in their

- core holder. By flushing solvents through the core it may be difficult to clean dead end pores.
- A change in temperature in the laboratory impacts the temperature of the detector which is in most cases very sensitive to temperature changes. If calibration scans are made at a different temperature than the experiment itself, it may lead to over- or underestimation of the water saturation. An easy way to correct for these effects is to use calibration scans outside of the core holder's position, for instance on a dummy core holder that does not change in time.
 - When performing X-ray scans for EOR (enhanced oil recovery) type of experiments, such as low salinity water flooding, complication is added to the interpretation of the attenuations. For example in a low salinity test, where we inject first high salinity brine, then followed by low salinity brine we notice that attenuation of high salinity brine (HS) is higher than that of low salinity brine (LS), so S_w cannot be calculated/measured during displacement of HS by LS when there is a mixture of HS and LS brine in the core. Change in X-ray intensity is the sum of the change in salinity and change in water saturation.
 - The X-ray measurement only measures fluid saturations along a line over the length of the sample and therefore samples only part of the core plug's volume (40% for a 1.5" sample). For heterogeneous cores, this induces an uncertainty.

We have used X-ray scanning to quantify the effect of low salinity water flooding in multiple experiments with crude/oil/brines from different fields. The concept behind low salinity water flooding is that more oil can be recovered from a core by injecting water that is *low in salt content* compared to the reservoir brine ((Tang and Morrow (1996), Berg *et al.* (2009), Masalmeh *et al.* (2013)). This is due to a changing interaction between the crude oil, formation brine and rock matrix. The exact mechanism is not completely understood, but possible mechanisms include double layer expansion (Ligthelm, 2009), and ion exchange (Lager, 2006). All these mechanisms lead to a wettability change towards water wet (wettability change is not the cause, but the effect). As the mechanism is unclear, the predictive capability is not there if one wishes to apply it in a real reservoir. Therefore one needs to perform laboratory experiments with the reservoir rock, the reservoir crude and the reservoir brine. In most cases a so-called tertiary flooding experiment is done where first high salinity (reservoir) brine is injected and then low salinity brine.

The recovery factor increase by injecting low salinity brine compared to normal reservoir brine is small, often between 0% and 10%. For a typical core plug, 1.5" in diameter and 5 cm in length, with a porosity of 30% and an initial water saturation of 0.3 this corresponds to 1.2 ml of oil.

Since it was unclear how to quantify the uncertainty in saturation as obtained using X-ray, and since typical volumes of extra oil were very small, an alternative method to determine the production of crude oil was designed.

EXPERIMENTAL SET-UP

We use the basic principle of the Welge set-up (Welge, 1952), see Figure 2. Water is injected from the bottom in the core using a Quizix pump (QX-20k) via a 1/16" diameter flowline (internal diameter 0.75 mm).

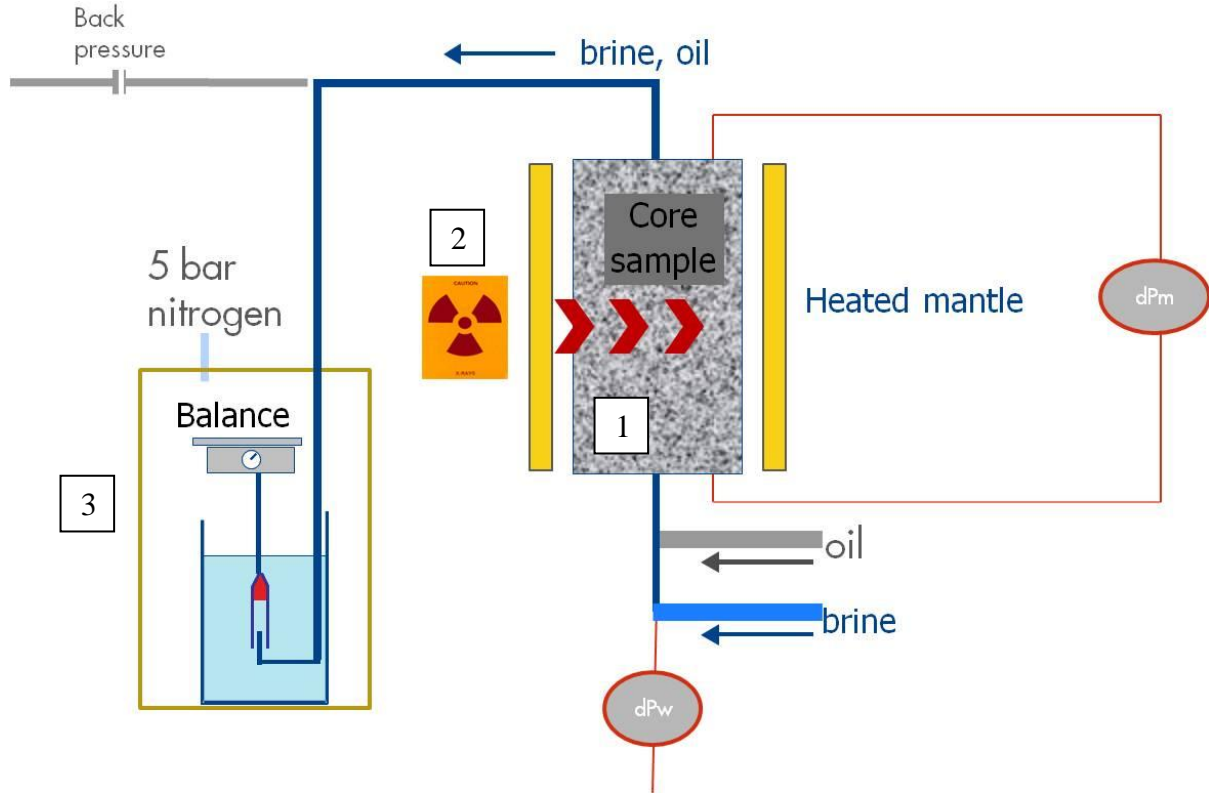


Figure 2 Schematic picture of experimental set-up, indication core + holder (1), X-ray source (2) and weighing unit (3).

The core is in a Hassler type of core holder. The output flow line (1/16" diameter) ends in a pressure cell that contains a balance (Mettler Toledo WK124-S). The pressure in the cell is kept constant using a pressure vessel filled with nitrogen. Below the balance, in the pressure container, we place a brine filled reservoir. The oil from the output flow line is produced in the brine filled reservoir, floats to the top and is captured in a cap that hangs from the balance. The volume of oil produced can be simply obtained from the reduction of the measured weight of this cap and the density difference between oil and brine:

$$V_{oil,produced} = \frac{F_{on_balance}}{(\rho_{oil} - \rho_{brine})g} - V_{inflow,dead} - V_{outflow,dead} = \frac{w_{on_balance}}{(\rho_{oil} - \rho_{brine})} - V_{inflow,dead} - V_{outflow,dead}, \quad (6)$$

where V is the volume of oil in m^3 , F is the force exerted on the balance in N, ρ is the density in kg/m^3 . By dividing denominator and numerator with g (acceleration constant $9.81 m/s^2$), we obtain the weight (w in kg) as recorded by the balance.

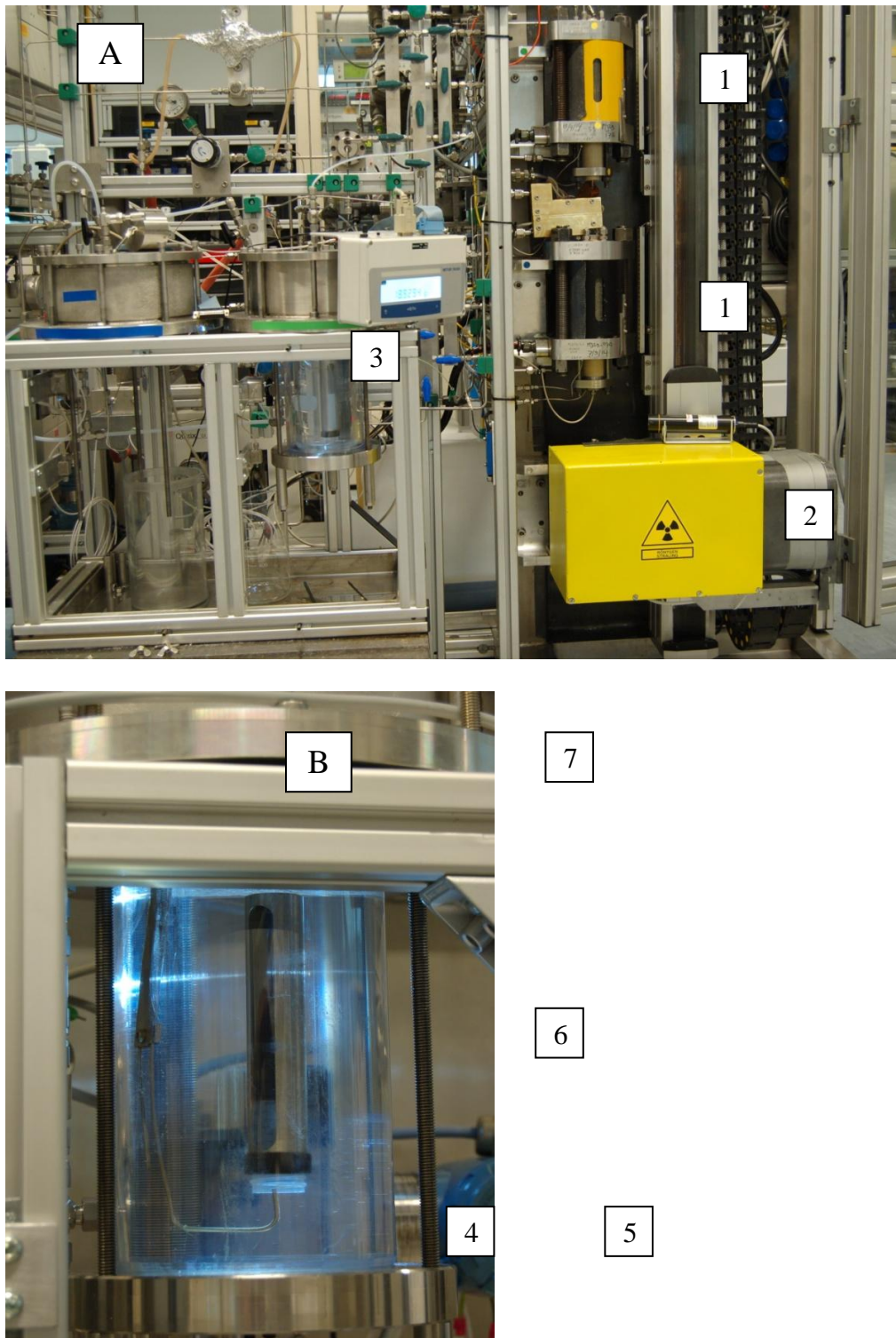


Figure 3 Experimental set-up with core holder (1), X-ray source (2) and weighing unit (3) (Picture A). Zoomed in picture of weighing unit showing the outflow flowline (4), the brine filled container (5) and the cap containing crude oil (6) hanging on the balance (7) (Picture B).

Uncertainties in the oil production come from the uncertainty in the density of oil and brine and from the uncertainty of oil in the inflow and outflow line (so called dead volume). When the brine injection starts, all flow lines are filled with oil. While injecting brine, the pressure drop over the core is monitored. The moment water hits the core, the pressure increases sharply and dead volume of the inlet flow line can be predicted accurately. The dead volume of the outflow flow line is determined by the volume injected between the point where first water is produced from the sample and the moment where first water is produced at the end of the outflow capillary. The latter is determined by the moment where the change in the weight on the balance starts to deviate from a linear relation which is related to oil production only. The first point where water is produced from the sample can be determined by either a jump in the pressure drop or a jump in the electrical resistivity over the length of the sample; this point cannot always be clearly identified. Dead volumes in the flow tubes are about 1.2 ml (inflow line) and 1.0 ml (outflow line). The total uncertainty from dead volumes is estimated to be less than 0.2 ml.

The main difference with a common Welge type of set-up (Welge, 1952) is that the balance, the aquarium and the cap are not at ambient pressure, but that they are at a back pressure of 5-6 bar. This adds a significant benefit: no gas is coming out of solution once the oil is produced. This would lead to interpretation problems as the density difference between air and water is much larger than between oil and water and thus a small volume of air will be erroneously measured as a significant volume of oil. This method can in principle be extended to reservoir conditions by using a magnetic coupling between production unit and the balance.

RESULTS

We show two examples where production from an unsteady state type of experiment is shown. In the first, production is inferred from our 'weighing-under-pressure' set-up corresponds very well with production obtained from X-ray. In the other experiment, they do not.

Figure 4 shows a low salinity polymer injection experiment at 50 °C and 6 bar back pressure. We used a sandstone rock in this experiment. At the beginning of the experiment, the core is at initial water saturation, filled with crude oil. First, we inject high salinity (formation) brine. After 145 pore volumes we increase the flow rate from 0.5 ml/min to 5 ml/min. More oil production is observed from the balance. The production from X-ray data decreases slightly, which is believed to be a temperature effect as the brine we inject may not be heated up completely at these high flow rates.

The difference between the two is less than 0.03 PV. After 450 pore volumes injected, we return to a flow rate of 0.5 ml/min and inject 1.8 cP low salinity polymer. More oil is produced. Production from X-ray and from the weighing unit match well within 0.01 PV.

We increase the flow rate in three steps to reduce any capillary end effect in the core and again, the match between X-ray and weighing unit is less, but still within 0.03 PV.

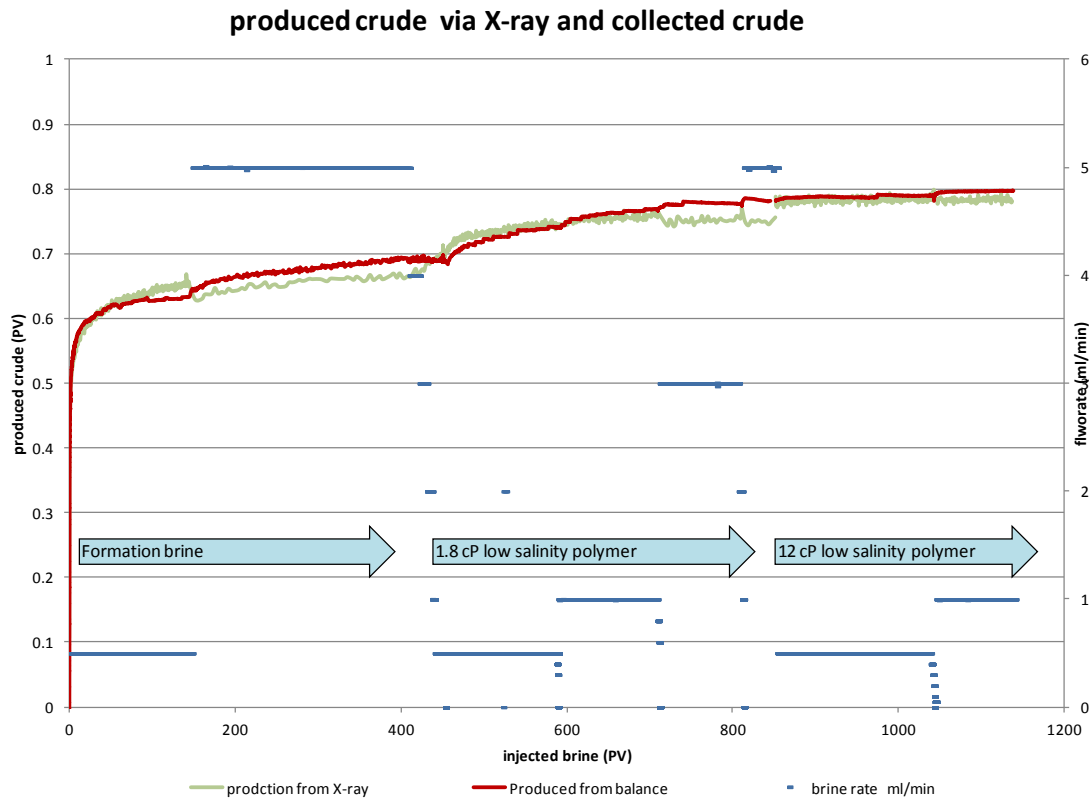


Figure 4 Water saturation calculated from direct production (red line) and calculated from X-ray absorption (green line). Sandstone sample.

As a last step (after 851 pore volumes injected), we inject a more viscous polymer solution (12 cP) and a few drops of extra oil are produced. Production from X-ray and weighing unit match within 0.01 PV.

In another low salinity water injection experiment, for a sandstone reservoir, we saw quite different results when we compared X-ray data with data from the weighing unit (see Figure 5). We start again injecting high salinity brine for 100 pore volumes, then switch to seawater. More oil is produced according to our new measuring method. The saturation from X-ray was already decreasing before switching to low salinity brine, and keeps on showing the decreasing trend after the switch. A decrease in saturation from X-ray is related to the lower salinity brine displacing the high salinity brine, but may also indicate precipitation of minerals in the core. After 170 PV's we switch to 5000 TDS brine, and again, extra oil is produced. The X-ray saturation shows a decreasing trend again because of the mixing of the brines. After 240 PV we inject 150 TDS brine, followed by a bumpflood. Again, increased oil recovery is seen from the balance (red line). At the end of the experiment, we switch again to the formation brine (high salinity

brine) which was the same as the brine we started with. It is interesting to see that the final saturation from X-ray is in good agreement with that from the weighing unit. This is not strange as the calibration scans are made at the end of the experiment, indicating that there is no dissolution/precipitation between the end of the experiment and the calibration scans. The overestimation of oil production at the first injection step with formation brine may be related to dissolution of solids during the experiment.

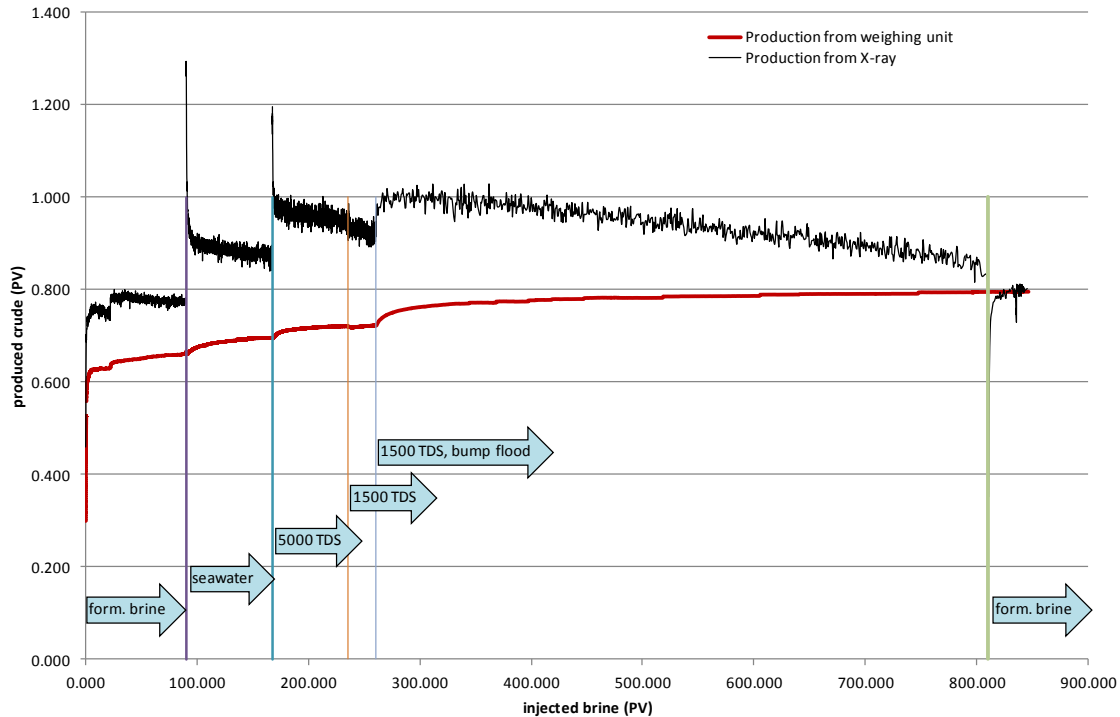


Figure 5 Water saturation calculated from direct production (red line) and calculated from X-ray absorption (black line). Sandstone sample.

We performed many experiments like the ones discussed and found that interpretation of X-ray saturations is not straightforward. Saturations were decreasing while they should be remaining constant or should be increasing, saturations were discontinuous when using different fluids, or initial saturations were not in agreement with initial water saturations obtained from porous plate or centrifuge. Especially for low salinity water flooding experiments, where contrasts between different fluids can be small, the interpretation of saturations from X-ray data is often not precise enough to be used alone to quantify the increased oil recovery by injecting low salinity brine. The ‘weighing-under-pressure method’ is much more precise and has less scatter. It serves as an excellent tool to quality check data obtained from X-ray. On the other hand, data from X-ray is useful as it allows one to track saturation profiles in the core, which cannot be done using the ‘weighing-under-pressure’ method. Therefore, a combination of the two techniques should be considered as a standard.

Although the uncertainty of the ‘weighing-under-pressure’ method is small, we still want to use a separate method to calculate in place volumes for quality assurance of the experiment. Instead of taking calibration scans after the experiment is done, we take the core out of the set-up and measure the water saturation using a Dean-Stark measurement. In Table 2 we show a comparison between water saturation at the end of an experiment using the weighing set-up and using Dean-Stark. The agreement between Dean-Stark and the weighing-under-pressure unit is very good. On average, the difference in volume is 0.25 ml, and the difference in saturation is 0.016.

One could opt to determine the final fluid saturation using Dean-Stark and then taking the calibration scans. In practice this would mean that the core has to be taken out of the set-up, and put back at exactly the same position to ensure that the same part of the rock is measured with the X-ray beam. This introduces another error and increases uncertainty. We have not investigated the magnitude of this uncertainty here.

Table 2 Comparison of saturations at the end of water flooding experiments, using the weighing set-up and using Dean-Stark. Difference in water saturation measured with the new weighing unit and Dean-Stark is 0.016 ± 0.015 .

Sample #	Brine volume in sample (ml)			PV (ml)	difference S_w
	from weighing set-up	from Dean-Stark	difference		
(SS=Sand Stone, Ca=Carbonate)					
SS1_1	12.3	11.5	0.8	13.6	0.06
SS1_2	9.9	9.9	0.0	14.6	0.00
SS1_3	8.8	9	-0.2	14.0	0.01
Ca1_1	15.2	14.9	0.3	20.7	0.01
Ca1_2	15	14.7	0.3	21.0	0.01
Ca1_3	15.8	16	-0.2	21.5	0.01
Ca1_4	14.3	13.9	0.4	18.8	0.02
Ca1_5	16.6	16.8	-0.2	21.0	0.01
Ca1_6	15.4	15	0.4	19.9	0.02
SS2_1	7.5	7.4	0.1	9.9	0.01
SS2_2	8.1	7.9	0.2	10.2	0.02
SS2_3	8.1	8.0	0.1	10.9	0.01
SS2_4	9	8.7	0.3	10.9	0.03
SS2_5	7.6	7.6	0.0	10.4	0.00

CONCLUSION

We have developed a new method to accurately determine the oil production from unsteady state experiments using a weighing unit under pressure. We have demonstrated that it is challenging to obtain reliable *in situ* water saturations from X-ray using undoped fluids. The uncertainty in X-ray saturation calculations include change of the rock matrix during the experiment, erroneous calibration scans on improperly saturated cores and mixing of injected fluids. The weighing-under-pressure unit is a new piece of technology that allows one to accurately measure the production of oil in unsteady state type of experiments without using dopants in the oil or the brine. This is especially a benefit in enhanced oil recovery experiments, where dopants could potentially influence the wettability of the rock. The volumes measured with our new set-up correspond well with the volumes inferred from Dean-Stark. We advocate using X-ray measurements in combination with the weighing-under-pressure unit to improve interpretation of flooding experiments, especially for low salinity water injection experiments or other type of EOR experiments. The weighing-under-pressure unit give precise production profiles, whereas the X-ray saturation profiles give valuable saturation profiles and information about the homogeneity of the core plug.

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