High-resolution inline density measurements: insight on multiphase flow and transport phenomena in porous media

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Abstract. An in-line densitometer is used in core flooding applications to quantify fluid production from core samples and obtain quantitative and qualitative information such as connate water production, breakthrough times, emulsion/foam generation, and steam condensation.

A series of core floods were performed with a densitometer placed at the outlet of a sandpack. All fluids passed through the measurement cell at experiential temperatures and pressures. The second series of tests were performed at high temperature and pressure, with a densitometer placed at the inlet and outlet of a sandpack, for steam applications. In both series of experiments, data acquisition was collected at 1 hertz and the analyzed density data was compared to results from the conventional effluent analysis, including Dean-Stark, toluene separations, magnetic susceptibility measurement, and flash calculations where applicable.

The high-resolution monitoring of effluent from a flow experiment through porous media in a system with two phases of known densities enables two-phase production to be accurately quantified in the case of both light and heavy oil. The frequency of measurements results in a high-resolution history of breakthrough times and fluid behavior. In the case of monitoring steam injection processes, reliable laboratory tests show that in-line density measurements enable the determination of steam quality at the inlet and outlet of a sandpack and qualitative determination of steam condensation monitoring

The use of in-line densitometry in core flooding applications provides insight on monitoring of complex fluid flow in porous media, which typical bulk effluent analysis is not able to do. The ability to measure produced fluids at high resolution and extreme temperatures reduces mass balance error associated with the effluent collection and broadens our understanding of complex fluid flow in porous media.

1.0 Introduction

Traditional coreflood systems utilize two/three-phase separators or fraction collectors downstream of the core to collect effluent. While phase separators prove reliable in conventional applications, they are limited in their ability to handle highly viscous samples and complex fluids like emulsions and nanofluids. Fraction collectors may cause an error in mass balance, due to misalignment of tubes, rate of sample entering tubes, movement of the collector's arm between tubes, and more. Additionally, fraction collectors result in low temporal resolution, as all effluent is mixed in the tube, resulting in an average composition over a given tube volume. Once the effluent is collected, analyzing it can be both time-consuming and challenging [1]. The dean-stark analysis is accurate; however, the temporal resolution is very low and, in many cases, only a single end-point oil production value is obtained. Methods such as NMR, solvent extraction, Karl-Fischer, and the like are costly, time-consuming, and limited to the resolution of the effluent collection method. In-line densitometry enables real-time quantification of produced water and oil and gives a valuable qualitative understanding of phase behavior and fluid flow in porous media.

A high-temperature and high-pressure density meter can be integrated into a coreflood set-up to measure effluent density and distinguish between crude oil and injection fluid. The method discussed involves weighing the produced oil to determine the weight of the residual oil remaining. The density measurements are used along with effluent analysis to facilitate laboratory studies of enhanced oil recovery.

Olsen et al. (2017) described a method for using a densitometer for quantifying oil production in two-phase coreflood experiments and looked at the understanding obtained by in-line density data for

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dynamic events such as water breakthrough, gas breakthrough, and connate water production. They conducted immiscible displacements at constant temperature and pressure conditions using North Sea light crude oil and were able to determine that the densitometer produced comparable data to that from an acoustic separator [2].

In this paper, we look at the potential applications and the challenges of utilizing in-line densitometry in core flooding. We demonstrate the benefit of using a high temporal resolution effluent analysis technique as a tool for understanding phase behavior and fluid flow in porous media. We look at the challenges of quantifying heavy oil and assess the use of in-line densitometry in emulsion, foam, and nanofluid core flood experiments. Additionally, we look at the applications of in-line densitometry for steam flooding and the potential understanding gained from being able to determine dynamic water saturation histories during hot gas flooding, condensation monitoring for steam flooding, and real-time volumetric steam quality data. Conventionally, acquiring such information is challenging and may require complex dynamic imaging techniques (i.e. CT, MRI) which are costly. This paper introduces easy and continuous methods using high-resolution densitometry for measuring and analyzing the data that cannot be obtained with conventional core flood configurations.

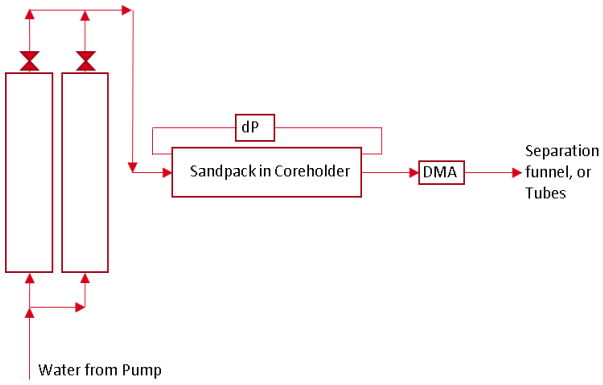


Figure 1: Simple schematic of multiphase core flood apparatus with the densitometer (DMA) located at the outlet of the porous media.

2.0 Methodology

The Anton Paar density meter (DMA HPM) operates on the principle of the oscillating U-tube, which has been used for over 50 years. The U-tube is excited by applying an electrical voltage to a Piezo element, causing it to oscillate at a characteristic frequency [3]. When fluid enters the measurement cell, the oscillation frequency is changed and is accurately detected by a second Piezo element. Each fluid has a characteristic frequency, which correlates to its density, as per the formula:

$$\rho = A\tau^2 - B \quad (1)$$

Where p is the density of the fluid, A and B are instrument constants, and τ is the oscillation period. The instrument constants A and B are determined in an instrument adjustment, where two reference standards, such as dry air and pure degassed water, are measured in order to determine the relationship between density and the period of oscillation. Using this relationship, unknown sample densities can be determined using their oscillation. The measuring cell is made from Hastelloy C-276, which can withstand high mechanical stress and is highly chemical resistant. The stated cell volume is 2 mL, however, the sensitive measuring volume is 0.35 ± 0.3 mL [2].

Multi-phase core floods were performed using a densitometer placed at the outlet of the sandpack. Density was measured at a frequency of 1 hertz and all measurements were made at the same temperature and pressure as the sandpack by using a circulating water bath and a back pressure regulator (BPR). The density meter was placed so that all fluid entering the sandpack passed through the measuring cell, before reaching the BPR and fraction collector. Flow rates used were < 0.35 mL/sec, in order to ensure that all fluid passing through the measuring cell was measured. If the flow rate exceeds the limit of the sensitive measuring volume, the accuracy of the analysis is affected.

Steam floods were performed in such a way that additives could be mixed with superheated steam resulting in wet steam laden with chemical additives that are present in the liquid phase of the injected mixture. These mixtures produced an increase in pressure drop relative to the back pressure of the apparatus which was then normalized against the pressure drops of steam of similar total heat values.

Table 1: The sandpack dimensions, geometry, permeability, porosity, composition, and pressure tap locations for steam foam experiments.

Dimensions	40.84cm X 2.244cm
Geometry	Cylindrical Unconsolidated
Permeability	2.27 Darcy
Porosity	40.14%
Composition of Media	Silica Sand 140-270
Tap Locations	Absolute Pressure at 0, 10cm, dP at 10, 20, 30, 40cm
Operating Pressure	200 psig
Operating Temperature	200 °C

The Experimental conditions for the steam foam floods were 200 °C 212 psia back pressure for the steam flood with four pressure taps, and 170 °C and 120 psia for the steam flood that only had pressure taps located at the inlet and outlet of the sandpack.

The dynamic saturation test was performed with nitrogen and water co-injected at a 4:1 gas to liquid ratio at varied flow rates to evaluate the equilibrium water saturation for a given flow rate at high-pressure high-temperature conditions. The back pressure of the system is adjusted in such a way that compressibility of flow is negligible and therefore unaccounted for.

Table 2: The sandpack dimensions, geometry, permeability, porosity, composition, and pressure tap locations for the dynamic fluid saturation experiment.

Dimensions	30.65cm X 1.5748cm
Geometry	Cylindrical Unconsolidated
Permeability	35.52 Darcy
Porosity	35.87 %
Composition of Media	Silica Sand 50-70
Tap Locations	Inlet, Outlet
Operating Pressure	750 psig
Operating Temperature	100 °C

2.1 Example Calculations

Olsen et al. (2017) determined a method for using in-line densitometry for quantification of produced water and oil in a two-phase system using the following equation [2]:

$$CumProdWater\ (0,N) = \sum_{0}^{N} \frac{MeasDens\ (i) - LowerEnvelope(i)}{Upper\ Envelope\ (i) - LowerEnvelope(i)} * [InjVol(i) - InjVol\ (i-1)] \quad (2)$$

Where the Lower Envelope is the density of the lightest phase, and the Upper Envelope is the density of the heaviest phase, oil and water, respectively. Alternatively, produced oil can be calculated directly using similar equation.

The produced oil fraction can then be determined by subtracting the produced water from the total injection volume

$$C(0, N) = CumInjFluid(0, N) - CumProdWater(0, N) \quad (3)$$

It is noted that the upper and lower envelopes must be carefully determined in order to ensure the accuracy of the analysis.

This equation can also be applied to miscible displacements, where the densities of the two pure phases are known.

In the case of highly viscous oil, the density measurement is dampened by a coating of oil on the walls of the measurement cell. This, in turn, lowers the apparent density. In this case, a moving upper envelope must be used. This is determined by applying a macro to the data set, which detects the change in the upper density value. If the change in density over a given produced volume is greater than a given value, in this case 0.0001 was selected as a cutoff, the upper envelope value is equal to the previously calculated value. This results in the upper envelope closely following the trend of the brine values, while accounting for slight fluctuations due to heavy oil coating the measurement cell, fines migration, fines settling in the measurement cell, changes in brine salinity, etc.

$$Moving\ Upper\ Envelope = IF\left(\frac{Measured\ Density\ (i) - Measured\ Density\ (i-1)}{CumProdVol\ (i) - CumProdVol(i-1)}\right) > 0.001, Upper\ Envelope\ (i-1), Measured\ Density\ Value \quad (4)$$

Liquid volumetric fraction calculation:

$$LVF = \frac{\rho Measured - \rho Gas}{\rho Water - \rho Gas} \quad (5)$$

Therefore, the vapor volumetric fraction can be assumed to be

$$VVF = 1 - LVF \quad (6)$$

The produced water/liquid can be calculated when the system pressure is significantly higher than the phase envelope pressure (boiling pressure) of the liquid phase. This is executed in discrete steps with each interval being each individual density measurement.

$$Produced\ Liquid\ per\ Data\ Interval = LVF * (Total\ Volume\ Produced)/(Unit\ Time) * Unit\ Time \quad (7)$$

Using the above equation, the cumulative produced liquid can be calculated as follows:

$$Cumulative\ Produced\ Liquid = \sum_0^N Produced\ liquid\ per\ Data\ Interval \quad (8)$$

Then using that information we can calculate the liquid saturation within the porous media as follows:

$$S_w = \frac{Cumulative\ Produced\ Liquid - Cumluative\ Injected\ Liquid}{Pore\ Volume} \quad (9)$$

For steam vapor, volumetric qualities are calculated in the same way as Equation (5)

The mobility reduction of steam foams is expressed as a mobility reduction factor.

$$Residual\ Resistance\ Factor\ (RRF) = \frac{Pressure\ Drop\ of\ Steam\ with\ Additive}{Equiblibirum\ Pressure\ Drop\ of\ Saturated\ Steam} \quad (10)$$

At steady state the equation for volumetric flow rate

$$Volumetric\ Flow\ Rate = (Mass)/(Unit\ Time) \times (Unit\ Volume)/(Mass) \quad (11)$$

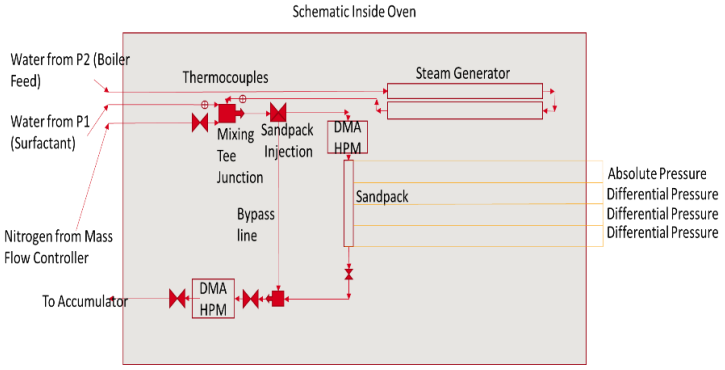


Figure 2: Schematic of the oven internals during steam flood/ hot gas flood experiments, the hot gas flood and the 170 °C steam flood only had one differential pressure measurement. The density measurement cell was inside the oven at 0.494 cubic centimeters of dead volume downstream the outlet of the porous media for all high-temperature experiments discussed in this paper.

3.0 Results and discussion

Here we summarized the capability of a high-resolution inline densitometer for flow experiments in porous media considering various condition including high temperature, high pressure, variable oil viscosity, and density differentials.

3.1 Oil recovery monitoring

The objective of this section is to show how we can precisely monitor the fluid production namely oil recovery in dynamic flow experiments.

The densitometer can be used to accurately determine initial oil in place (OOIP). Additionally,

mass balance errors associated with the effluent collection and analysis techniques are eliminated by providing in-line results. Figure 3 to Figure 5 shows the densitometer results for conventional displacing experiments with Dodecane as the oil phase.

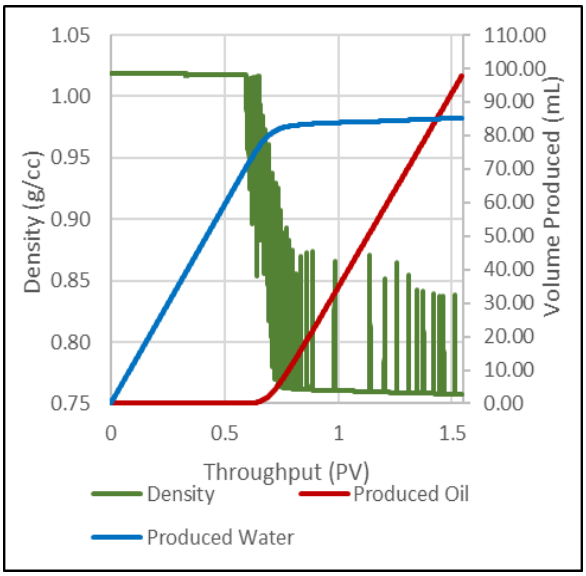


Figure 3: Oil displacing water in water-saturated sandpack. Density monitoring for determination of original oil in place.

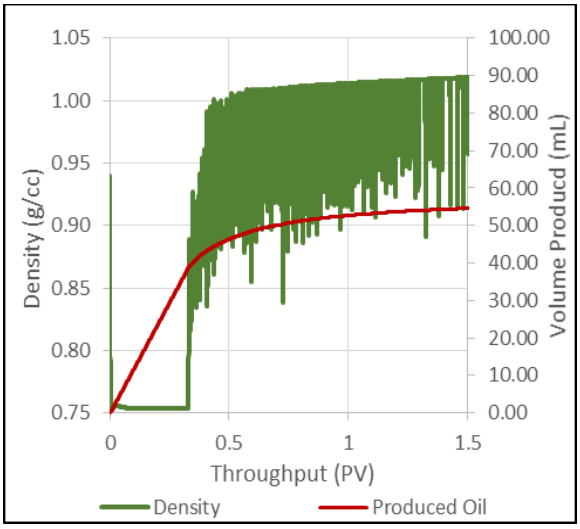


Figure 4: Density history of waterflood through Dodecane saturated sandpack.

As shown in Figure 4, the high-resolution monitoring of effluent from a flow experiment through porous media in a system with two phases of known densities enable oil production to be accurately quantified. The frequency of measurements results in a high-resolution history of breakthrough times and fluid behavior.

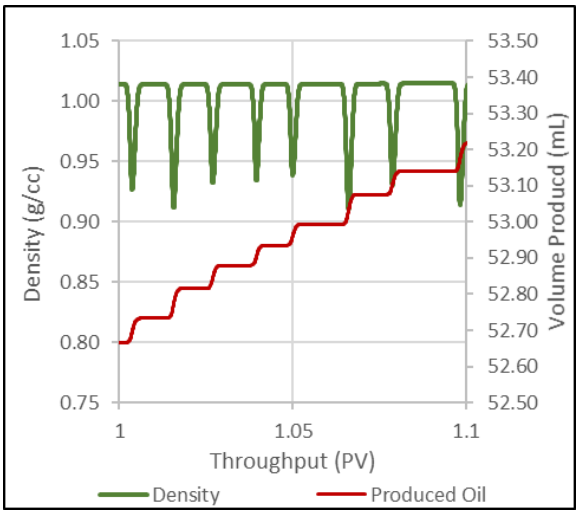


Figure 5: A closer look at density history of waterflood through Dodecane saturated sandpack

A closer look at the density history enables us to identify that the oil is being produced in "trains" (Figure 5) rather than as an emulsion, which is similar to the observation made by Olsen et al. [2]. This is because the density levels off at the density of the water phase, based on the calibration, before dipping down towards the density of the oil. If the oil was being produced as emulsion, there would be smaller oscillations in density, at a density representative of the quality of the emulsion.

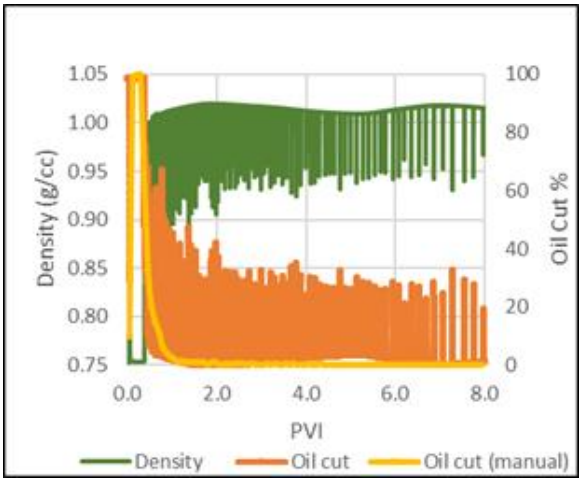


Figure 6: Comparison of density calculation to manual determination of oil production.

Here we demonstrate that the densitometer provides reliable results when compared to effluent fraction collection and analysis. The mass balance from the effluent analysis was 99.6% and the mass balance from calculated throughput analysis of the densitometer was 100.2%. The mass balance can be

improved by utilizing a circulating water bath in the measurement cell of the densitometer, which was considered in the next section. As shown in Figure 6, effluent analysis cannot accurately capture the oil production performance especially after 2 PV. On the other hand, the oil cut from density calculation shows that the oil is still producing even after 8 PV of the injection. The change in amplitude of the density curve provides us information about the volume of oil production. The change is the frequency of the density will tell us how often oil is producing. For example, in Figure 6, the frequency of density after 5 PV is lower than that of the early time stage (1-2 PV) supporting the fact that the oil production rate decreases after 5 PV.

3.2 Heavy oil recovery calculation; potential and challenges

As shown above, using the density meter for oil saturation allows for a quick determination of original oil in place. This is more crucial when working with heavy crude oil. The effluent analysis is difficult with heavy oil, as it coats the collection vessel and makes visual determination inaccurate. Solvent separations are also time-consuming.

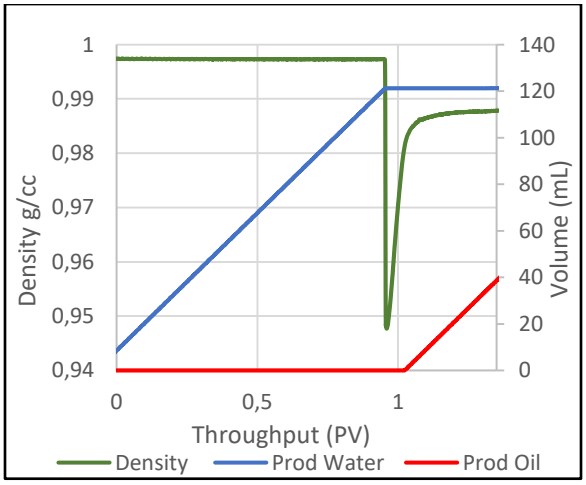


Figure 7: Density history of heavy oil saturation in water saturated sandpack

To verify the effectiveness of oil saturation with the density meter the effluent was passed through the measurement cell and collected into a seporatory funnel. The OOIP was 111 mL from analysis of the seporatory funnel and 113 mL by the densitometer. The negative throughput is due to dead volume production from the lines first passing through the measurement cell for both Figure 7 and Figure 8. The percent relative difference between the two methods was 1.79%.

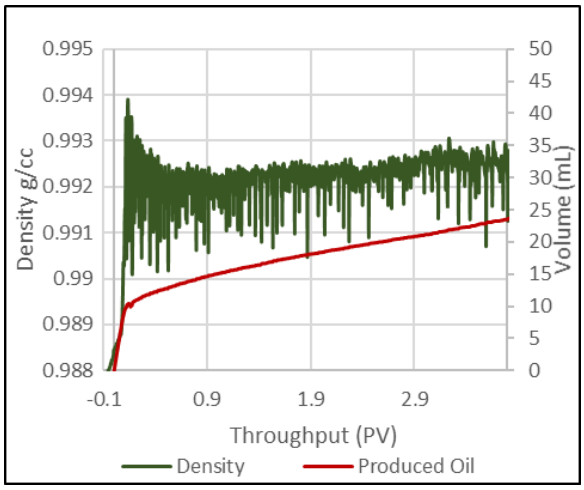


Figure 8: Density history of waterflood through heavy oil saturated sandpack

The quantification of heavy oil through the densitometer is challenging, due to its tendency to coat the walls of the measurement cell. However, if the upper envelope (UE) is adjusted accordingly (See equation 3- Moving upper envelope), it is possible to obtain accurate quantitation of heavy oil production. In order to verify the densitometer results, the effluent was collected into a seporatory funnel and analyzed. According to the manual method, 23.6 mL of oil was produced. According to the density meter results, 24.8 mL of oil was produced. The methods had a percent relative difference of 5.1%. It should be mentioned that several core flood tests with heavy oil were performed and the average percent relative difference was ± 10%. This error, which is due to heavy oil coating the measurement cell, could be reduced by heating the measurement cell to lower the oil viscosity and reduce coating effects. Further development of this method is required before it can be used as a stand -alone analysis of heavy oil production. However, the density meter shows potential for elimination of challenging effluent analysis, including the need for time-consuming Dean-Stark analysis.

3.3 Nanoparticle transport in porous media

There is a linear relationship between the density and concentration of nanofluid, enabling the densitometer to be used as an inline analyzer for quantification of nanofluid in a two-phase system. Errors could arise from adsorption of stabilizing agent to the porous media and fines migration that can be potentially

detected by high-resolution density measurement. It should be mentioned that fine migration can occur in different tests and may result in additional errors.

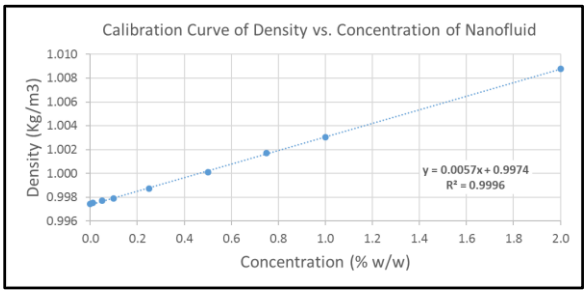
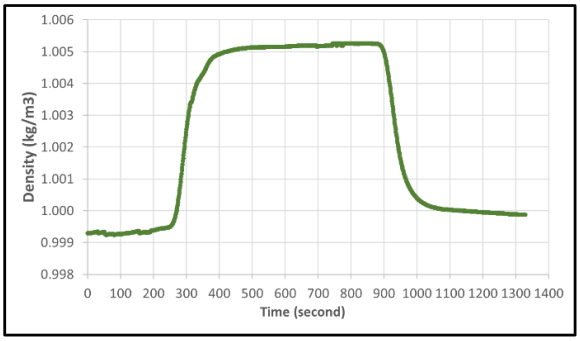


Figure 9: The density of silica nanoparticle at different concentrations. High-resolution density measurement allows accurate detection of nanoparticle with a relatively small increment of nanoparticle concentration.

Following is an example of densitometer capability to detect nanoparticle transport in porous media. The results were also correlated with the magnetic susceptibility measurement of the effluent samples. A suspension of iron oxide nanoparticles was injected in a water-saturated porous media (properties of porous media is mentioned in Table 2) at room condition. Thereafter, the nanofluid displaced with post flush of water. The iron oxide nanoparticles have magnetic properties so can be detected by magnetic susceptibility measurement. The magnetic susceptibility is normalized based on the magnetic susceptibility of effluent without iron oxide nanoparticles (Figure 10 b) any value above zero shows the presence of iron oxide nanoparticles. More detail on such experiments and magnetic susceptibility measurement can be found elsewhere [4].

The normalized magnetic susceptibility profile shows that the particle breakthrough occurred around 250-300 seconds. However, the density profile can accurately show the breakthrough time (260 seconds) since it is a continuous measurement with 1 Hz frequency.



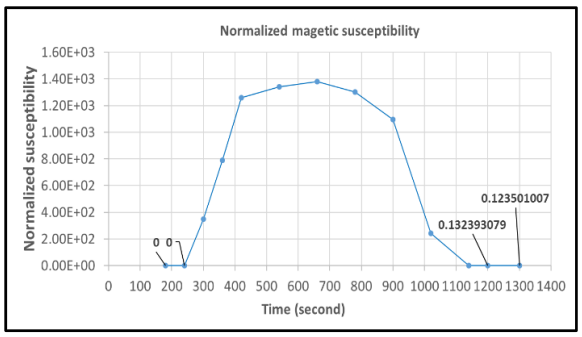
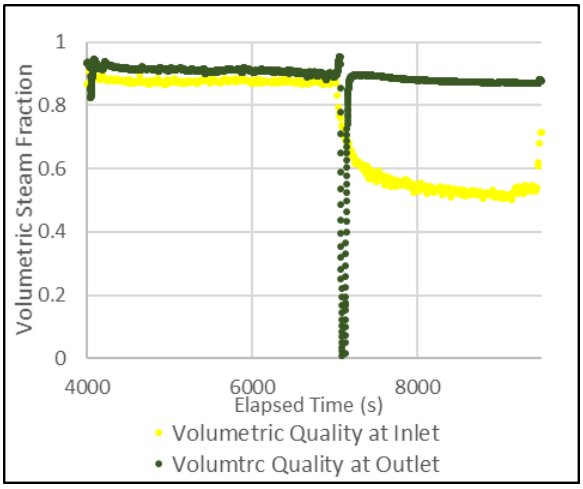


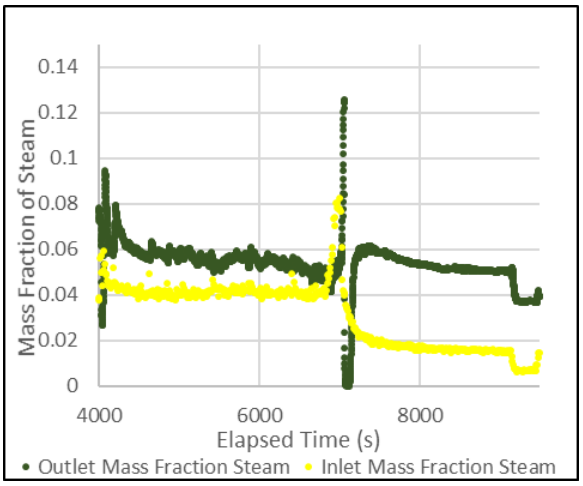
Figure 10: Density profile (a) and magnetic susceptibility profile (b) of iron oxide nanoparticle showing the breakthrough time and nanoparticle transport behavior. Post flush of water started at 600 seconds.

Interestingly the density profile at 1200-1300 seconds is higher than the beginning (water density) suggesting the nanoparticle are still presents in the effluent sample. The dispersed iron oxide nanoparticles in water create a very dark color liquid which is easily detectable with the naked eye even in low concentration (e.g. 0.05 wt %). The effluent sample at 1200 and 1300 seconds in Figure 10b was very clear by naked eyes, but the susceptibility measurement suggests that there is still nanoparticle in porous media supporting the density results. It is worth mentioning that monitoring transport of nanoparticles in the presence of a second fluid (i.e. oil) will add more challenges especially if the nanoparticles remain at oil-water interface.

3.4 Monitoring saturated steam and hot gas multiphase flow through porous media

Steam/saturated steam flow in porous media presents an especially complex multiphase flow scenario where densitometry can provide some qualitative and semi-quantitative data. High-resolution densitometry enables us to monitor –but not quantify- steam condensate production of the porous media, and we observe distinct variances during foam production as it compares to wet steam flow. Volumetric steam qualities can also be calculated from the density histories, and consequently, mass qualities are also able to be calculated when assuming the system is at steady state.





Figures 11: Monitoring volumetric (a) and mass (b) steam quality (inlet/outlet) with the help of inline densitometer. An ambient oven temperature of 170 °C was used.

Steam foam flood baseline is from 4000 seconds to 6850 seconds as shown in Figure 11. This flood had a flash calculated steam quality of injection of 0.4-0.6, however, the flash calculation does not account for the heat losses associated with the steam moving through the stainless steel tubing while being injected into the porous media. Densitometry enables the analyst to have experimental data to determine what the true steam qualities of injections are. In this particular scenario, the mass flow rate of the steam was 10 g/min and the thermodynamic flash calculations provide a mass quality result of 50% +/10% while the density meter results are less than 10% mass quality for both the inlet and the outlet. The density meters placed at the inlet and outlet, also enables us to observe that in this particular experiment, the outlet mass quality is higher than at the inlet due to flashing off of liquid as the absolute pressure decreases during propagation through the porous media.

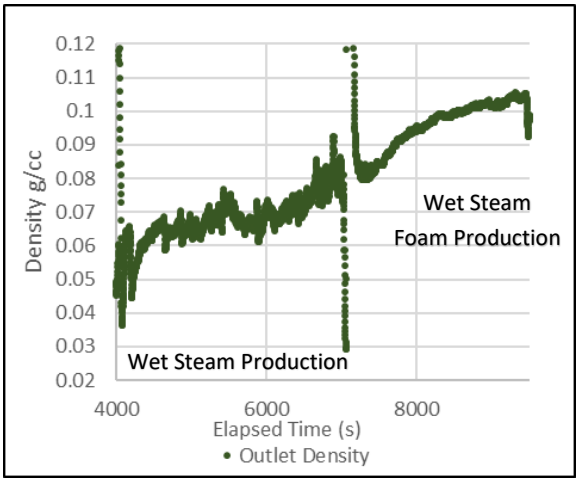


Figure 12: Foam/no-foam boundary identification for steam foams at the outlet of porous media. An ambient oven temperature of 170 °C was used.

Figure 12 shows the steam (4000 seconds to 6850 seconds) and steam foam flood experiment. The steam foam flow has a distinctive characteristic compared to the pure steam flow at the same steam quality. Steam foam production has a distinct lack of random fluctuations that are observed in the saturated steam production.

Steam condensation can be monitored with the density profile (Figure 13). This will enable us to evaluate the breakthrough times of steam and find out when vapor began being produced at the outlet.

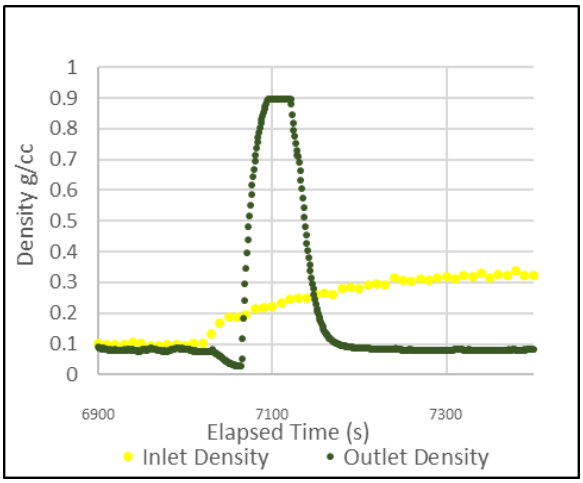


Figure 13: Steam condensation monitoring with the help of inline densitometer.

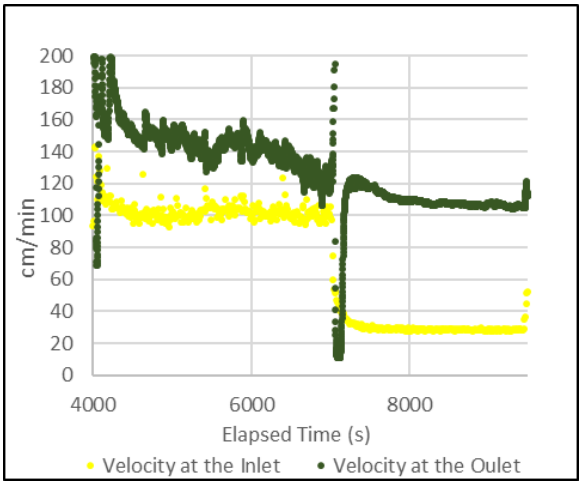


Figure 14: The true velocities of steam moving into the porous media. Steam flood (baseline) is from 4000 seconds to 6850 seconds. An ambient oven temperature of 170 °C was used.

The true velocities of steam cannot accurately be calculated with flash calculations and heat loss modeling. Figure 14 demonstrates the capability of calculating volumetric flow rates during steam flooding. This is not possible to calculate accurately without the use of densitometry due to the complex nature of the heat transfer that is involved with steam floods such as oven convention speeds, insulation, etc. Calculating the velocity based on flash calculations results in a falsely high velocity. The calculations all assume the system in is steady state.

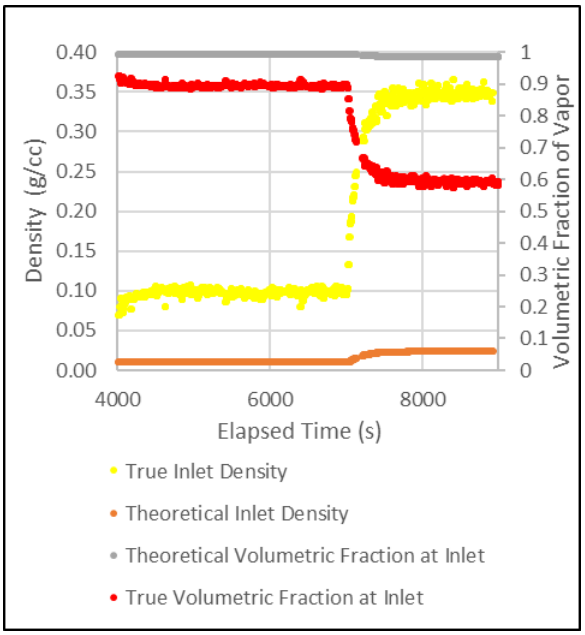


Figure 15: A comparison of the theoretical calculated density and volumetric fraction of the steam being injected into the porous media, highlighting the comparison between thermodynamic estimations of steam volumetric quality and measured steam volumetric quality using the DMA HPM density meter.

The true velocities of the being injected into the porous media can readily be calculated using the stream density and the mass flow rate which provides us with accurate flow velocities of steam. Using the density meter has enabled more accurate predictions of velocities than with flash calculations alone due to real time measurements of density, or specific volume of the injected phase as highlighted in Figure 15. The instrument also provides a without a doubt assurance of what phase of state the injected fluid is in which aides greatly in steam foam floods since occasionally rapid changes in pressure cause condensate production. In some cases, it's helpful to troubleshoot whether a chemicals performance is simply due to the lack of the presence of a steam phase (instrument limitation), or to distinguish that a chemical simply does not perform in the presence of the condensable gas phase.

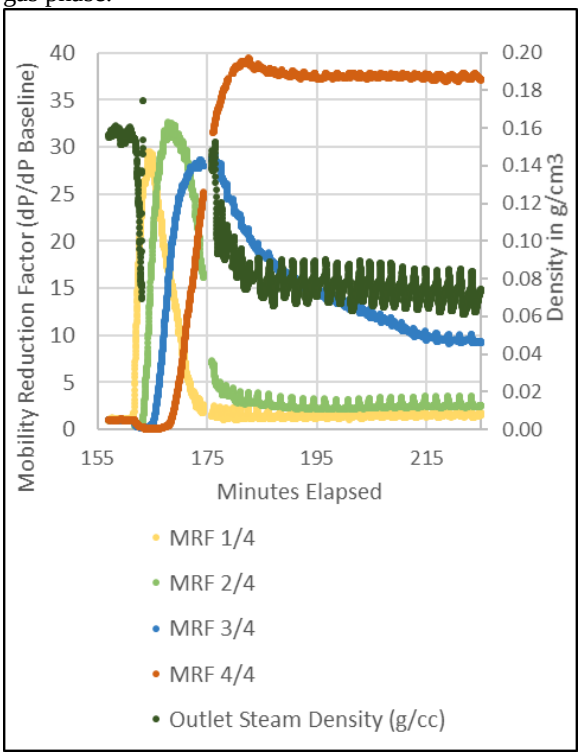


Figure 16 mobility reduction factor and density profile during steam foam injection. The baseline was performed from 130 minutes until 165 minutes, which is only partially visible in this plot. This steam flood was performed at 200

°C with the outlet at the saturation pressure regulated with a back-pressure regulator.

As shown in Figure 16, initially we see the equilibrium steam density at baseline. The mixing water is then forced to stop mixing with the superheated steam prior to injection and is substituted by a specially designed foaming solution. Immediately a reduction in density occurs simultaneously to the increase of differential pressure in the first quarter of the sandpack. As resistance to flow increases the bubble point temperature of the steam also increases, this means that heat must be delivered to the porous media in the form of latent heat which generates condensate. We also observe the true breakthrough of the steam phase, which normally would be assumed to be at the maximum differential pressure of the last section of the sandpack, however, we can observe that steam actually begins to form at the maximum differential of the 3rd quarter of the sandpack. This is caused by the flashing of the condensate and should not be considered the actual breakthrough time of the injected steam front.

The ability to dynamically measure liquid saturation allows us to analyze the equilibrium water saturations during multiphase flow conditions as shown in Figure 16.

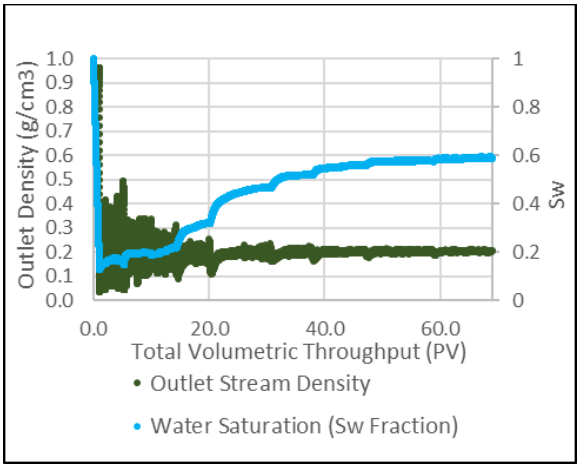


Figure 17: A plot demonstrating the use of densitometry to calculate dynamic fluid saturation. This test was performed at 100 °C and 750 psig back pressure. Total Volumetric flow rates were 0.5, 1.0, 1.5, 3.0, 6.0, 10.0, 15.0, 20.0 28.0 mL/min respectively

3.5 Quality Control of Dynamic Fluid Saturation

It should be noted that beyond ~21cc/min does not provide the density meter with enough time to measure "every molecule" that passes through it, the residence time within the DMA HPM is not long enough however the saturation values still aligned quite closely with manual mass balance performed by weighing the isolated sandpack before and after the flood. Manual mass balance results in a water saturation of 0.497 in comparison to the 0.587. The relative difference between the two methods is 16.6%. According to manual mass balance 10.66 mL of water was left within the sand pack, and according to the DMA HPM 12.56 mL of water are left inside the sand pack, this error could be due to the fact that once velocities above ~21cc/min were used at the last two velocity intervals the materials flowing through the DMA HPM did not have enough residence time to be accounted for in the cell. The error is magnified in the S_w calculations because of the reduced pore volume, a larger pore volume would reduce the impact of these uncertainties. The total volume (of water) injected was 281.67cc, the total volume produced was 281.08cc. A mass balance deficit of -0.2% is obtained. The DMA HPM has proven to be an effective tool for mass balance for two-phase flow based on the data obtained. The deficit is only due to the flow rates being slightly higher than the maximum flow rate at which all material can pass through the measuring cell and have a density measurement associated with it.

4.0 Summary

Understanding fluid flow in porous media is challenging. Laboratory experiments at real reservoir condition are key to achieve this goal. However, the nature of experiments (pressure, temperature, fluid viscosity, multiphase flow, etc.) makes the data analysis and interpretation challenging and sometimes impossible. This study introduces the high-resolution inline density measurement at experimental condition (i.e. high pressure, high temperature) as a unique tool for understanding fluid flow in porous media. Following are some of the unique achievement of data analysis utilizing densitometer (DM):

- Beside oil recovery calculation in conventional light oil experiments, DM can significantly reduce the time, chemical usage for oil recovery calculation in tests including heavy viscous oil without compromising the accuracy.

- Valuable information in high-temperature experiments including monitoring steam condensation, steam/steam foam propagation and breakthrough time, water saturation changes, and steam front velocity calculation.
- Understanding complex fluids flow by identifying foam/no foam boundary at reservoir condition without the need for visualization.
- Monitoring and detecting nanoparticles or fine transport in porous media.

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