

Carolina de Barros Bruno

Instituto Superior Técnico
September, 2014

Abstract

It is an established fact that a substantial amount of oil remains in the reservoir(s) after the application of primary and secondary methods. Therefore, there is currently a huge incentive for the development of appropriate EOR methods designed to recover a portion of the remaining oil. The correct choice of one or more methods depends on extensive knowledge of the petrophysical characteristics of the reservoir rock, the presence of fluids and the interactions between the various components in different phases of the production cycle.

In this master thesis were determined unconsolidated siliciclastic rock absolute and effective permeabilities, at room temperature and pressure, using two glass tubes developed for the purpose. Were also assessed the effective permeability changes that occurred during the test, when a surfactant was employed to support the oil extraction.

The absolute permeability tests were conducted in vertical and horizontal directions, using deionized water or brine with 35g/L NaCl injected in sandstone sandpacks. In both directions, the absolute permeability values varied between 0.4 and 9 mD and the curves of the water effluent versus the time experiments were similar. When the brine was drained through the sandpacks, very different permeability values were obtained, ranging between 28 and 1mD. In the effective permeability tests was employed iso-octane, having its extraction failed due to the high interfacial tension prevailing in the upper part of the sandpack. The use of AOT surfactant allowed to recover 12.8% of the initially injected hydrocarbon.

Considering the experimental results, it is important to continue the effective permeability tests with the surfactant employment at different concentrations in order to increase hydrocarbons recovery rates.

Keywords: Absolute permeability, effective permeability, unconsolidated sandstone sandpacks, iso-octane, deionized water, brine.

1. Introduction

According to Archer & Wall (1994), a reservoir may be defined as an accumulation of hydrocarbons in a porous permeable sedimentary rock, being common, in a single oilfield, the presence of several reservoirs in stratigraphic horizons or different pressure regimes. The setting for hydrocarbons accumulation is a sedimentary basin that has provided the essential components for petroleum reservoir occurrence: (a) a source; (b) the formation and migration; (c) a trapping mechanism, i.e., the existence of traps in porous sedimentary rock at the time of migration and on migration path.

The reservoirs behavior is governed by viscous, capillary and gravity forces leading, stabilizing or limiting the fluids movements in its interior and their distribution depends on rock geological characteristics, rock-fluid properties, flow mechanisms and production forms.

The first stage of hydrocarbon recovery involves the use of the natural energy stored in the reservoir. As the reservoir pressure dissipates, the oil flow is directed to producing wells and

can be increased when water is injected through injection wells (secondary recovery). When the water ratio necessary for pumping oil and gas becomes too high, the process is finished. However, after the primary and secondary recovery operations, more than two thirds of the oil initially present in the reservoir (OOIP), still remains. This is due not only to the residual oil high viscosity which limits its mobility, but also to high interfacial tensions between hydrocarbons and liquid phases which result in higher capillary forces retaining oil in smaller pores (Gudiña, 2013). Hence, the maximum amount of hydrocarbons extraction is one of the biggest challenges in oil industry (Alvarado et al, 2010). Trapped oil recovery usually involves the use of expensive tertiary recovery methods.

The correct choice of EOR method and the exact calculation of the oil remaining amount in the reservoir depends upon detailed knowledge of the reservoir rock petrophysical characteristics – porosity, permeability, capillarity and wettability – the existing fluids and the interaction between the various components in the different phases of production cycle.

Porosity can be defined as the pores volume in the rock, which control the amount of fluids contained in the reservoir. The effective porosity only considers the pores that are connected together, thus allowing the fluid passage.

Additionally to Darcy's law, capillarity is another important physical phenomenon in flow through porous media. The coexistence of immiscible phases at the pore level results in curvature at the interface between any two phases. There is a tension between any two phases due to the dissimilarity of the intermolecular forces between the molecules comprising the phases. These molecular interactions create a force that tends to contract interface in a smaller area, which creates a tension that is dependent only on the presence of this interface (Li, 2011).

The fluid distribution in the pore space is affected not only by the fluid/fluid interface forces, but also by the forces of the fluid/ solid interface. Wettability is the tendency of one fluid to spread on or adhere to a solid surface in the presence of a second fluid. The reservoir rocks can be categorized as water-wet, intermediate-wet or oil-wet (Gomes & Alves, 2011).

Permeability measures the porous medium ability to transmit fluids and it can be absolute or effective. Effective permeability is the ability to preferentially flow a particular fluid when other immiscible fluids are present in the reservoir. In contrast, absolute permeability is conducted when a single fluid, or phase, is present in the rock.

Absolute permeability can be measured in a laboratory by direct or indirect methods. The first ones can be classified into four categories: those using gas or liquid; under steady-state or unsteady-state (transient) conditions. Permeability can be inferred by empirical correlations in indirect measurements, such as those of inter granular porosity and grain size (Carman and Kozeny); pore-throat and distribution of the grains through mercury injection or measuring capillary pressures (Swanson, Katz and Thompson, etc.); effective electrical conductivity (Archie, Ondracek); transmissivity of the acoustic waves (Biot), among others. Usually these methods are more time consuming and less accurate than the direct ones (API, 1998).

Axial flow steady-state method using incompressible liquids, provided the basis for the proposed experimental procedure in order to measure absolute permeability. This method has several advantages, including:

- (1) Pressure differentials with liquids are higher and easier to measure than those with gases;

(2) No gas slippage correction is required, and;

(3) Accurate permeability measurements with liquids may be more representative of reservoir permeabilities.

Relative permeability is the ratio between effective permeability of a particular fluid at a particular saturation, and absolute permeability of that fluid at total saturation. This property can be measured by means of two methods: steady-state or unsteady-state conditions (Dandekar, 2006). In the steady-state method, fluids are injected into the core or sandpack sample at a fixed ratio until pressure and saturation equilibrium are reached. Relative permeabilities are calculated directly from the flow rate and pressure drop data. The unsteady-state method involves injecting a single fluid in a core or sandpack sample at either constant rate or constant pressure while measuring the amount of fluid displaced and the pressure drop or rate, respectively, with time. Relative permeabilities are then calculated using either the method of Johnson *et al* (1959); or a reservoir simulation method (Archer & Wong, 1973) or a regression method (Sigmund & McCaffery, 1979). More recently, Watson *et al.* (1988) have developed an improved regression-based method that is believed to honor the experimental data more closely. The actual displacement process in the reservoir is an unsteady-state displacement, as such the unsteady-state method more closely reproduces fluid flow in the reservoir (Morton-Thompson & Woods, 1997).

Relative permeabilities measured in laboratory can be affected by several parameters, including:

(1) Selection of the rock sample: This property analysis generally requires the use of homogeneous samples.

(2) Test conditions - temperature and confining pressure: According to Hawkins (1989) using crude and higher temperatures do not change the relative permeability curves, but may affect the residual oil saturation during secondary recovery. In the oil reservoir, the rock is subject to a net overburden pressure, which is equal to the weight of the vertical column of rock and fluid minus the pore pressure of the rock. This property is inversely proportional to the permeability, i.e. the higher the pressure overburden, the lower the permeability.

(3) Wettability: Wettability has been shown to have a large impact on relative permeability. According to Morton-Thompson & Woods (1997), the water relative permeability curves for water-wet rock is higher in an oil-wet rock; this is because in an oil-wet rock, the water is in the center of the pores. By flowing through the center of the pores, water has a less tortuous path, hence a higher effective permeability.

(4) History saturations: When measuring relative permeability, it is important to consider the saturation history. Many researchers have observed relative permeability hysteresis. Hysteresis can mean that relative permeabilities obtained are different in a waterflood than in an oil flood (flooding phase hysteresis). Relative permeabilities are sometimes different the second time a sample is water flooded than the first time it is water flooded (cycle-dependent hysteresis). Thus, laboratory relative permeability measurements should mimic reservoir flooding history. The water injection data should be measured in the first cycle of flooding.

Objectives

The present work aims to observe the changes in absolute and effective permeability in a set of unconsolidated sandstones sandpacks, on vertical and horizontal directions, and evaluate the interactions between the various system components. For this purpose were designed and built two devices called glass flow tubes with capacity to hold the sandpacks and allow the injection of fluids at constant rate. The experiments were performed with deionized water, brine with 35g/L NaCl dissolved and isooctane, which were applied either together or individually.

In the effective permeability experiments, measured following the unsteady-state technique, was also intended to obtain the residual water and oil saturations and to assess the effective permeability changes with the addition of 150 mM surfactant Dioctyl sulfosuccinate sodium salt dissolved in the hydrocarbon.

2. Proposed methodology and materials

The proposed methodology is divided in three parts: (1) absolute permeability tests in vertical direction; (2) absolute permeability tests in horizontal direction and (3) effective permeability testes in vertical direction.

2.1. Materials used

2.1.1. Rock matrix

The siliciclastic rock chosen to reproduce the reservoir rock was composed of partially cemented fluvial sandstones from moderate depths (fluvio-deltaic sediments) of Formação da Lourinhã and was collected in Praia d' El Rey. This rock was chosen due to its geological similarities with reservoir rocks of Brazilian petroleum systems.

The dynamic sandpack flow tests were performed with a set of disaggregated rock sandpacks. The sandpacks samples preparation began with the manual homogenization of the sandstone rock and its division into smaller fractions by quartering. The subsamples obtained were subsequently divided using a Jones type splitter and then weighed.

The sandpacks granulometric composition was 20,4% gravel, 70,8% sand, 2,5% siltes and 6.3% clays and the average porosity ranged between 25 and 32%.

2.1.2. Fluids

In the absolute permeability experiments was used deionized water and brine composed by 35g/L NaCl dissolved in deionized water and in the effective permeability tests was additionally used isooctane and an anionic surfactant.

Isooctane is an organic compound named by IUPAC as 2,2,4 – trimethylpentane . It was selected not only because it is a long chain hydrocarbon and its structure resembles hydrocarbons compounds present in reservoirs but also due to its known chemical composition that allows the tests standardization.

Dioctyl sulfosuccinate sodium salt from Sigma-Aldrich commonly known as AOT was the selected anionic surfactant for the experiments. It was used to reduce the water surface tension. AOT is more soluble in less polar solvents like hydrocarbons.

2.2. Experimental apparatus and procedures

2.2.1. Permeability tests in vertical direction

The sandpacks absolute permeability tests were conducted following the axial flow, steady-state method (API, 1998) in a flow tube developed and built for the purpose.

The flow tube is made of glass and has 4 cm inner diameter. At the tube bottom was placed a sintered glass (G3) that acts as a filter (15 and 40 micra), allowing not only to retain the rock sample but also to prevent the smaller particles loss during the dynamic flow experiments. The flow tube is connected to the positive displacement pump by a polyamide tube, which is fixed to the flow tube by a 4cm inner diameter glass lid with a glass adapter. Two taps were incorporated to the flow tube to allow the samples desorption pre-treatment with a vacuum line.

The experimental apparatus consisted of: a glass tube; a positive displacement pump; a technical balance; a vacuum line and a computer with RS232 connection to the balance and WinCT software Riskey version.



Absolute and effective permeability experimental apparatus in vertical direction

2.2.1.1. Absolute permeability experimental procedures

When using new and dry sandpacks it was proposed the following procedure:

First, the sandpack (subsample) was placed inside the clean tube until a pre-defined calibration (100mL), followed by compaction of the subsample through impact by manually tapping the tube 40 times on a hard surface. After compaction, the subsample level was checked and more sample was added to complete the pre-defined volume.

Then the tube was connected to a vacuum line and the desorption treatment took place during one hour. The connection between the vacuum line and the tube was made through the tube bottom tap.

After the pre-treatment, the sandpack was soaked with deionized water and on the sandpack top there should exist a supernatant water that remains approximately constant during the experiments. A positive displacement pump was employed to maintain liquids injection at a constant rate. On the bottom of the tube, it was placed a recipient to weight the effluent. The effluent mass measurements were accomplished at intervals of 60 seconds during one hour. The following expression was applied to calculate the absolute permeability:

$$k = \frac{C_2 \cdot C_4 \cdot q \cdot \mu \cdot L}{C_1 \cdot A \cdot \rho \cdot g (h + L)}$$

When a previously soaked sandpack was reused (formerly used in a test) , it was only required to connect the tube to the pump to restart the procedure.

2.2.1.2. Effective permeability experimental procedures

2.2.1.2.1. Isooctane

For the effective permeability tests were employed sequential injections of deionized water or brine and isooctane, using the same experimental apparatus. It was applied the following procedure:

In a previous soaked sandpack, isooctane was pumped. To obtain the isooctane effective permeability, were collected effluent samples, at intervals of 10 min, using graduated cylinders of 10mL to determine the liquids fractions volumes. The deionized water was dyed with copper sulfate to increase the contrast between the phases - the water and the hydrocarbon.

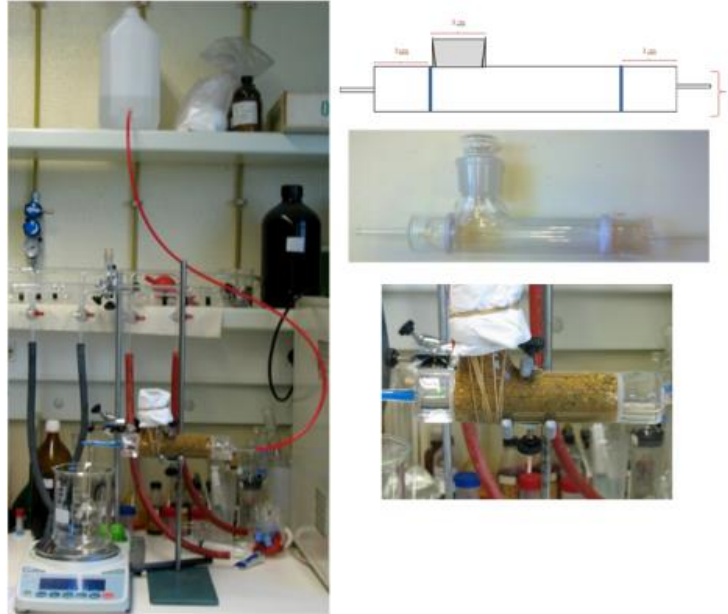
2.2.1.2.2. Isooctane with surfactant

In order to change the interfacial tension between water and isooctane, the AOT surfactant was dissolved in the hydrocarbon and pumped through a previously soaked sandpack with deionized water. The following procedure was applied:

The mixture of 150mM AOT in isooctane was injected in a 100% soaked sandpack with deionized water. The mixture was weighed on a technical balance at intervals of 10 minutes and each liquid fraction volume was determined in graduated cylinders of 25 and 10mL.

2.2.2. Permeability tests in horizontal direction

To carry out tests in the horizontal direction, it was necessary to adjust the flow tube, the injection regime and consequently change the experimental procedure. The second flow tube was constructed with a 4 cm inner diameter and two sintered glasses (G3) placed at both ends. They allow not only to retain unconsolidated sandstone sandpacks inside the tube, but also to obtain a laminar flow during the experiments. On one side was added a ground-glass to ease the subsamples placement inside the tube.



Absolute permeability experimental apparatus in horizontal direction

2.2.2.1. Absolute permeability experiments procedures

When using new and dry sandpacks, it was proposed the following procedure:

The sandpack (subsample) was firstly placed inside a clean tube until it was completely filled, followed by compaction of the subsample through impact by manually tapping the tube 40 times on a hard surface. After compaction, the subsample level was checked and more sample was added to complete the tube.

To establish a liquid injection constant flow , it was installed a 5L container 60cm above the flow tube and both were connected by a polyamide tube. At the flow tube bottom it was placed a glass recipient to enable the effluent weighing. The effluent mass was measured at intervals of 60 seconds during one hour. The following expressions were applied to calculate the absolute permeability:

$$k = \frac{C_2 \cdot q_f \cdot \mu}{C_1 \cdot G_f \cdot (p_1 - p_2)} \quad G_f = \frac{\pi \cdot D I^2}{4L} \quad p_1 - p_2 = \rho g \Delta h$$

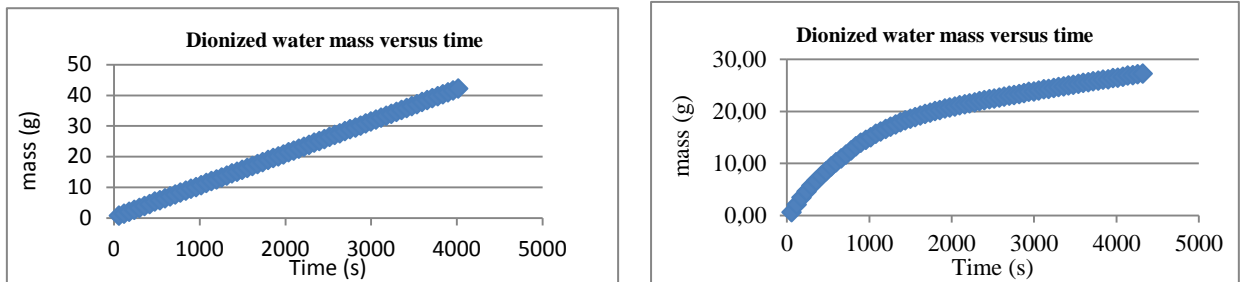
When a previously soaked sandpack was reused (formerly used in a test) , it was only required to recharge the 5L container and restart the procedure.

3. Results and discussion

3.1.1. Absolute permeability experiments in vertical direction

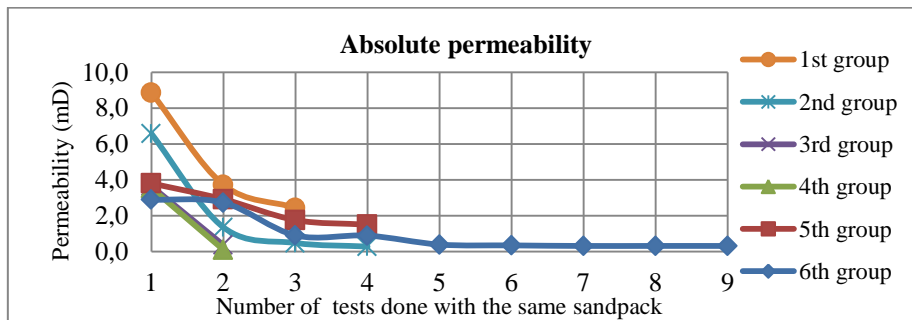
25 tests organized into six groups were performed, in the first four groups the sandpacks were dried between each test, while in the two last groups (5 and 6), the sandpacks remained soaked with water. The absolute permeability values obtained ranged from 0.4 and 9mD. The curves of the water effluent versus the time experiments were similar in the first four groups, however a change has been observed in the curves when the sandpack remained fully soaked (5 and 6 group) between successive tests. This effect may be related to the smaller particles leaching

from the sandpacks. As such, to calculate the the 5th and 6th groups absolute permeability, were only used the instantaneous flow rates obtained after the flow reached the steady-state regime (after 1000s).



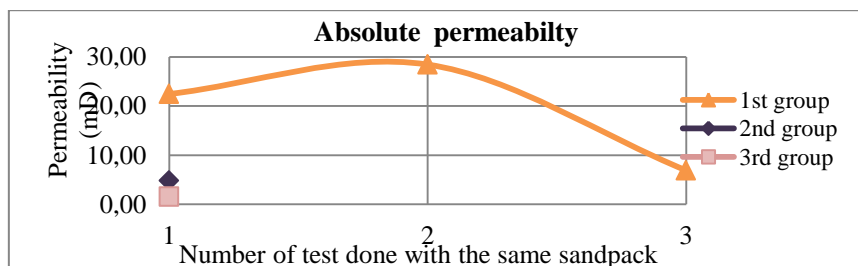
Deionized water mass versus time curves of the 1st group second test (on the left) and of the 6th group second test (on the right)

The absolute permeability decrease in the tests with deionized water may be related to the release of exogenous particles in suspension that can migrate, plugging the pores and thus reducing sandpacks permeability. The release of particles from the surface may be caused by two different phenomena: (1) chemical effect or "water sensitivity of sandstone" and/or (2) the hydrodynamic effect. The first phenomenon occurs when siliciclastic sandpacks are in contact with fresh water and it is due to the clays swelling, its migration or a combination of these two effects. The second phenomenon relates to a mechanical effect induced by the fluid hydrodynamic forces, the latter generates a less severe permeability decline than the chemical effects because only a few particles are released.



Absolute permeability experimental results in the vertical direction using deionizad water

The absolute permeabilities obtained in tests with brine were very different ranging from 28 to 1mD. Due to this large gap, it was not possible to conclude which effect – chemical or hydrodynamic – has greater influence on sandpacks.



Absolute permeability experimental results in the vertical direction using birne

3.1.2. Effective permeability experiments in vertical direction

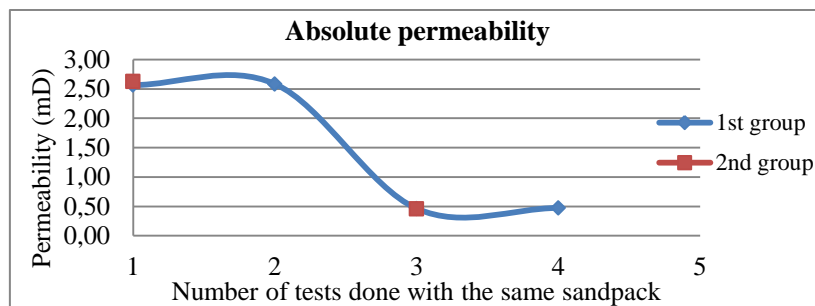
Four effective permeability tests were accomplished, three with deionized water and one with brine. In the first experiments the isooctane was pumped in fully soaked sandpacks with water or brine and in last test the sandpack was dried before the isooctane injection. The hydrocarbon extraction failed in all the tests, even when the pore space saturation conditions and the type of fluid pumped were altered. Both water/brine as well as isooctane were retained in the sandpacks because the viscous or gravitational forces in the pore space were insufficient to overcome the capillary forces.

Due to the failure, it was decided to test the isooctane injection in a dry and new sandpack. It was verified that the isooctane goes through the porous medium, not being trapped. This phenomenon can be explained by the sandpacks geological characteristics which absorb water – possibly being water-wet.

Only with the use of AOT surfactant was possible to recover 12.8% of the initially injected hydrocarbon. The oil flow through the sandpack may have been due either to interfacial tension decrease between water and oil or to the sandpack wettability alteration from a water-wet to a mixed-wet or even to a combination of both.

3.1.3. Absolute permeability experiments in horizontal direction

In all the experiments done in horizontal direction, the sandpack remained soaked between successive tests. The absolute permeability values obtained ranged from 0.4 to 2.5 mD and the curves of the water effluent versus the time experiments were similar to the horizontal direction tests.



Absolute permeability experimental results in the horizontal direction using deionized water

It is proposed the alteration of the liquid entrance and exit locations in the tube, to limit the presence of huge air volumes that renders difficult the water flow.

4. Conclusions and recommendations

In this thesis dynamic flow experiments were accomplished to study the absolute and effective permeability changes in sandstone sandpacks at room temperature and pressure, horizontally and vertically, in order to evaluate the interactions between the various system components. Were also assessed the effective permeability changes that occurred during the test, when a surfactant was employed to support the oil extraction.

The absolute permeability values varied between 0.4 and 9 mD and the curves of the water effluent versus the time experiments were similar in both directions. Unlikely when brine was used, very different permeability values were obtained, ranging between 28 and 1mD.

In all permeability effective experiments, the hydrocarbon extraction failed, even when the pore space saturation conditions and the type of fluid pumped were changed. Both water/brine as well as isooctane are retained in the sandpacks, this is due to the high interfacial tension prevalent in the upper part of the sandpack. The use of AOT surfactant allowed to recover 12.8% of the initially injected hydrocarbon. This phenomenon may have been due either to interfacial tension decrease between water and oil or to the sandpack wettability alteration from a water-wet to a mixed-wet or even to a combination of both.

To complement the results presented in this dissertation, it is important to continue the effective permeability tests with surfactant employment at different concentrations in order to increase hydrocarbons recovery rates. Since most hydrocarbon reservoirs have saline adjacent aquifers, we propose the development of tests with the surfactant injection in previously soaked sandpacks with brine.

5. References

- Alvarado, V. *et al* (2010) – Enhanced Oil Recovery: An Update Review. 2010, 3, 1529-1575; doi: 10.3390/en3091529
- API (1998) – Recommended Practices for Core Analysis. Recommended Practice 40 Second Edition. Exploration and Production Department
- Archer, J.S. & Wall C.C. (1994) – Petroleum Engineering. Principles and Practice. Graham & Trotman.
- Dandekar, A. (2006) – Petroleum Reservoir Rock and Fluid Properties. Taylor & Francis. pgs 223-226
- Guidinã *et al* (2013) – Biosurfactant-producing and oil-degrading *Bacillus subtilis* strains enhance oil recovery in laboratory sandpack columns. Journal of Hazardous Materials 261 (2013) pgs. 106– 113
- Gomes, J.S & Alves, F. (2011) – O Universo da Indústria Petrolífera, da Pesquisa à Refinação. Fundação Calouste Gulbenkian. Serviço de Educação e Bolsas.
- Li, Robert (2011) – Study of Foam Mobility Control in Surfactant Enhanced Oil Recovery Processes in 1-D, Heterogeneous 2-D, and Micro Model Systems. Doctor of Philosophy, Texas.
- Morton-Thompson & Woods (1997) – Development Geology Reference Manual: AAPG Methods in Exploration Series, no. 10. Pgs 226-228