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On Effects of Wettability on Multiphase Flow in Porous Media

by

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A THESIS

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Abstract

Heavy oil resources have become an important sector of oil and gas industry. The Athabasca oil sand deposit, mainly in the McMurray Formation, is the largest and most important one in Alberta, Canada. The wettability of porous media is the most important property that directly controls multiphase flow and phase distribution. But there are still arguments about the wettability of the McMurray Formation oil sands and factors that affect the wettability and the consequent water-oil relative permeability curves. In the research reported in this thesis, surface mined unconsolidated McMurray Formation oil sands were analyzed for its wettability and effect on relative permeability. A new method to change the wettability of oil sands sand grains was developed and the wettability was explored by examination of contact angle and relative permeability. It was found that asphaltene and resin adsorption or precipitation on the surface of sand grains alter their wettability from water-wet to oil-wet. Furthermore, water displacement tested are performed to estimate the impact of wettability of the sand grains on the relative permeabilities of oil and water calculated by unsteady state method. The results suggest that in steam chambers where the oil has been extracted at elevated temperature, there is potential for a change of the wettability of the oil sands.

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I would like to thank my kind supervisor Dr. Ian Gates for his support, guidance, encouragement and understanding during my study. I would also like to thank Mr. Jacky Wang for his continuous help and valuable suggestions during my research.

Dedication

I dedicate this dissertation to my husband Sheng Yang, my daughter Kaylee Luxi Yang and my parents for all their wholehearted devotions to carrying me to this stage.

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Chapter One: Introduction

1.1 Introduction

Unconventional hydrocarbon resources have become an important sector of the oil and gas industry. Heavy oil or bitumen is one type of unconventional petroleum resource, which makes Canada one of world's largest hydrocarbon resource countries. The most important feature of heavy oil and bitumen is their high viscosity (100-50,000 cP for heavy oil and 100,000+ cP for bitumen) under original reservoir conditions. Heavy oils in Alberta are mainly found in the Lloydminster area whereas bitumen is mainly deposited in the Clearwater and McMurray Formations in the Cold Lake and Athabasca deposits, respectively.

Generally, most sandstone reservoirs are typically considered water-wet. However, the exposure of crude oil to the rock can change rock wettability (Anderson, 1986a). The composition of heavy oils has hydrocarbon components that have larger molecular weight than that of conventional oil, which makes the effects of heavy oil on the wettability of the rock more complex. Thus, determining reservoir rock wettability and understanding heavy oil and bitumen effects on sand grain wettability are a fundamental requirement for design of recovery processes.

Water flooding is a commonly applied secondary recovery method. Reservoir rock wettability directly affects multiphase flow, which is described by relative permeability curves. Determining the relative permeability curves with respect to the state of wettability is a crucial step for understanding the performance of water flooding and improving its recovery factor.

This purpose of the research documented in this thesis is to: 1. determine the wettability of an unconsolidated McMurray Formation oil sand, 2. development of a method to alter the wettability of the sand, and 3. evaluate the impact of wettability of the sand grains on the relative permeabilities of oil and water.

1.2 Heavy Oil and Bitumen Resources in Alberta

There are three main oil sand deposits in Alberta: the Athabasca deposit, the Cold Lake deposit, and the Peace River deposit. The viscosity of the bitumen in these deposits is over 100,000 cP. The Athabasca oil sands deposit has the largest initial volume in place whereas the Peace River deposit has the least one. The porosity of these oil sand reservoir ranges from 18% to 33% (Alberta Energy Regulator, 2015) and the oil saturation is between 60 and 90%. In terms of permeability, the average horizontal permeability of the Athabasca and the Cold lake oil sand reservoirs are around 4 D and 2.2 D, respectively (Gates, 2013).

The high permeability of these reservoirs is favourable for oil production. However, the high viscosity hinder the oil from flowing and practically, no oil flows from the reservoir when the oil is at its original reservoir conditions. However, temperature strongly affects the viscosity of the oil. Figure 1.1 shows the viscosity is significantly reduced when the temperature is raised; its viscosity is under 10 cP when the temperature is greater than 200°C. Thus, thermal recovery methods, where the temperature is raised to over 200°C, are feasible recovery processes for oil recovery from these systems.

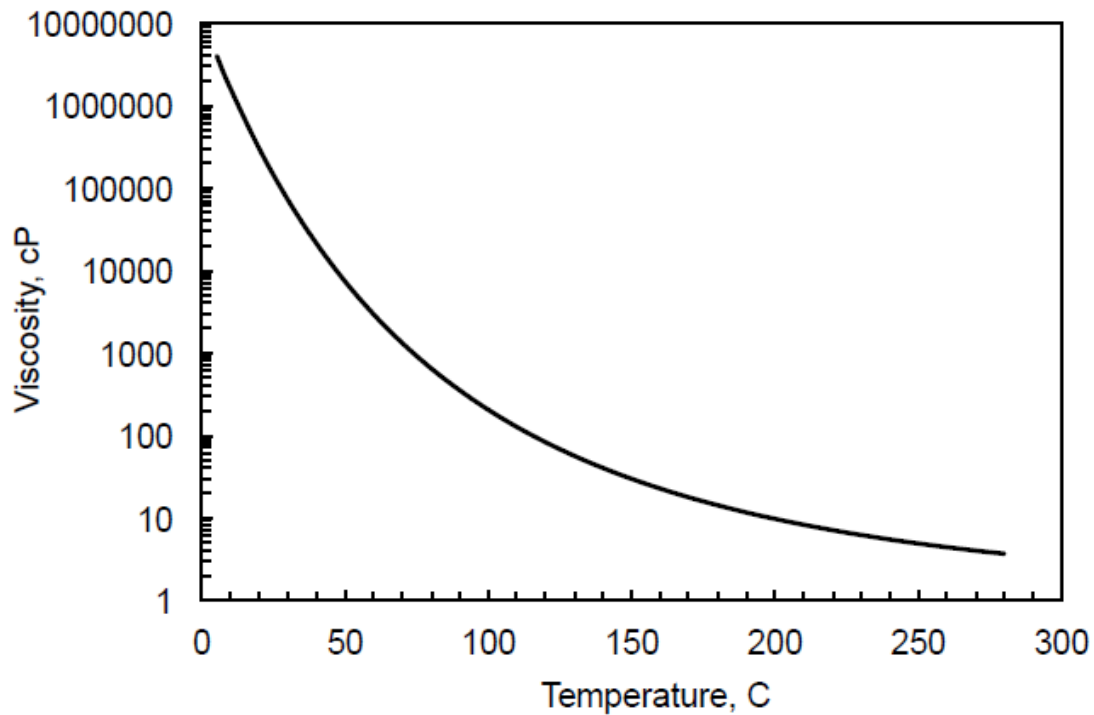


Figure 1.1 The relationship between viscosity of Athabasca and temperature (Mehrotra and Svrcek, 1986).

1.3 Recovery Processes for Heavy Oil and Bitumen

Cold production and thermal recovery are the two main method for heavy oil and bitumen production, respectively. Cold production mainly refers to primary recovery for heavy oil where it flows under its own energy. The main mechanism of cold production is pressure difference driven where solution gas drive and oil foam formed by gas exsolution as the pressure drops when the well is put on production (Tremblay et al., 1999). The cold production flooding process has been applied in Lloydminster heavy oil reservoirs (Adams, 1982) with recovery factor ranging from 2% to 8% of original oil in place (OOIP). Subsequent water flooding processes of heavy oil

reservoirs can only increase 1% to 2% of OOIP. Cold production with sand (CHOPS) is an enhanced cold production method where sand is also produced from the reservoir. Under CHOPS process, the recovery can reach 15%. One big problem is modelling formation of wormholes for design of cold production recovery processes (Istchenko and Gates, 2014).

The main thermal recovery processes include cyclic steam stimulation (CSS) and steam-assisted gravity drainage (SAGD). Both processes have been applied in oil sands reservoirs, while the high cost of generating steam still limit their economic outcomes.

CSS is also referred to as the “huff’n’puff” process. The same well is used as both the injection and production well. In the first step, high pressure steam is injected into reservoir. In the second step, injection is stopped and the hot zone ‘soaks’ within the reservoir further heating more of the reservoir. In the third step, the well is opened to production and mobilized oil, steam condensate, and gas is produced from the reservoir. One important feature of CSS is that the injection pressure is usually higher than the formation fracturing pressure. This means that shale layers in reservoirs are not a big barrier for fluid flow since they are broken by steam fracturing (Bao et al., 2016).

SAGD consists of a upper injection well and lower production well each separated by about 5 m with the production well positioned typically about 2-3 m above the base of the oil sands reservoir. After steam heating the formation, SAGD mode is conducted by injecting steam into the upper well and mobilized oil, steam condensate, and gas is produced from the lower well. The important features of SAGD are the formation of the steam chamber within the formation and continuous oil drainage under the action of gravity. Compared with CSS, the injection pressure of SAGD is

relatively low. As a result, the effects of solution gas and formation recompression are low and since steam fracturing is not done, shale layer can significantly block steam and heat transport.

1.4 Relative Permeability for Heavy Oil and Bitumen Reservoirs

Relative permeability is among the most important rock-fluid properties since it directly impacts water and oil flow within the porous medium, and consequently the oil production rate and ultimate recovery factor. It is thus one of the most important parameters affecting reservoir simulation models. In terms of heavy oil and bitumen systems, both cold production and thermal recovery are significantly affected by relative permeability among water, oil and gas. Most research on relative permeability for heavy oil and bitumen reservoirs focus on the effects of temperature, and there is a controversy about its effects. Rock wettability is a crucial factor controlling multiphase flow and their relative permeability curves in conventional reservoirs (Anderson, 1986a; Anderson, 1987b). However, there are only a few studies of effect of wettability of relative permeability in heavy oil and bitumen systems under a constant room temperature. This is the subject of the research reported in this thesis.

1.5 Organization of Thesis

Chapter 2 reviews the literature about wettability, methods of wettability measurements, wettability alteration, relative permeability measurements and effects of wettability. Chapter 3 describes the experimental equipment setup and basic measurements of rock fluid properties. Chapter 4 presents the preparation of oil-wet and water-wet sands. Chapter 5 describes the water-

flooding experiments for water-wet and oil-wet sands and the evaluation of relative permeability.

Chapter 6 lists conclusions and recommendations resulting from the research in this thesis.

Chapter Two: Literature Review

2.1 Wettability

Wettability directly affects the electrical properties of porous media (Anderson, 1986c), capillary pressure (Anderson, 1987a), multiphase flow and phase distribution (Anderson, 1987b) and water flooding performance (Anderson, 1987c). Craig (1971) defined wettability as “the tendency of one fluid to spread on or adhere to a solid surface in the presence of other immiscible fluids.” In oil reservoirs, the wettability measures the rock’s or sand grain’s preference for oil or water. For example, when the rock has a strong preference to water, it is referred to as water-wet and water tends to spread or adhere around the sand grains. Similarly, when the rock has a strong preference to oil, it is oil-wet and oil spreads or adheres around the grains. As a consequence of wettability, the more strongly wetting phase tends to occupy the smaller pores of the rock and the non-wetting phase occupies the more open pores, as illustrated in Figure 2.1.

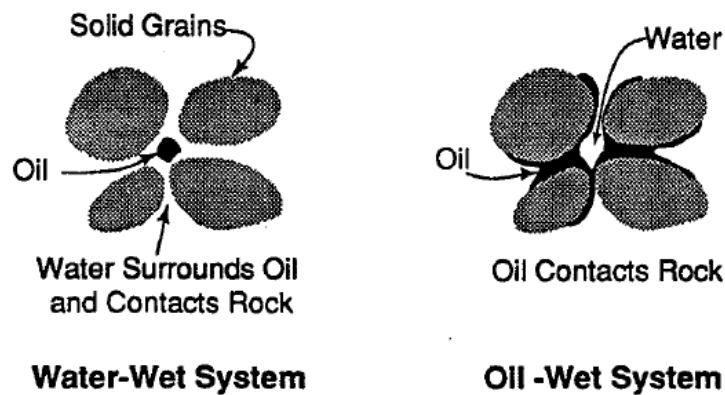


Figure 2.1 Fluid distributions in water-wet and oil-wet systems (Green and Willhite, 1998).

When sands grains have no strong preference for either oil or water, the system is termed as having neutral/intermediate wettability. In other situations when different parts of rock have different fluid preferences, this system is referred to as having fractional wettability (Brown and Fatt, 1956). Most hydrocarbon reservoirs are believed to be strongly water wet; before oil migrated into the reservoir rock, the rock was likely filled with water (Anderson, 1986a). Many carbonate reservoirs and a few sandstones reservoirs have been shown to be oil-wet or fractional wettability (Katz, 1942; Treiber and Owens, 1972; Chilingar and Yen, 1983; Xu and Dehghanpour, 2014).

2.2 Methods of Wettability Measurement

Quantitative and qualitative methods are two major categories of wettability measurement. Quantitative methods include contact angle, Amott method, and the U.S., Bureau of Mines (USBM) method, and qualitative methods include imbibition, microscope examination, flotation, glass slide, relative permeability curves, capillary pressure curves, capillarimetric method, displacement capillary pressure, permeability/saturation relationships, and reservoir log techniques (Anderson, 1986b).

2.2.1 Contact Angle

The contact angle is the most applied method in oil industry (Anderson, 1986b). Many methods have been applied to measure contact angle. The sessile drop is generally used in the petroleum industry. In this method, the rock surface is thoroughly cleaned and immersed in oil. Next, a drop

of water is placed on the rock surface as shown in Figure 2.2. The contact angle, ranging from 0 to 180° is measured.

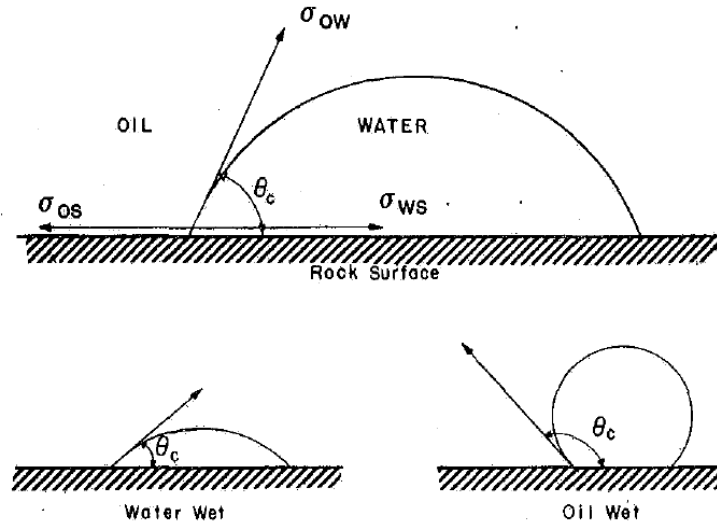


Figure 2.2 Contact angles of water-wet and oil-wet systems.

The Young's equation has been used to represent the surface energies in the systems (Adamson and Gast, 1967) as follow:

$$\sigma_{ow} \cos \theta = \sigma_{os} - \sigma_{ws} \quad \text{Eq. 2-1}$$

where the contact angle, θ , is generally measured through water, σ_{ow} is the interfacial energy (interfacial tension, IFT) between oil and water, σ_{os} is the interfacial energy between oil and solid (rock), and σ_{ws} is the interfacial energy between water and solid (rock).

When the contact angle (through the water phase) is between 0 and 75°, the system is considered as water wet. When a contact angle (through the water phase) is between 105 to 180°, the system

is called oil wet. When the contact angle is from 75 to 105°, the system is considered as neutrally wet. The literature reveals different cut-off values to describe these three states of wettability but in general, these values are typical cut-off values for defining the wettability of the rock.

The adhesion tension is given by (Amyx et al., 1960):

$$\sigma_A = \sigma_{os} - \sigma_{ws} = \sigma_{ow} \cos \theta \quad \text{Eq. 2-2}$$

When the adhesion tension is positive, the system is water wet. When the adhesion tension is negative or near 0, the system is oil wet or neutrally wet, respectively. There are some limitations of contact angle measurements, including hysteresis, heterogeneity of rock surface and presence or absence of organic coatings on the surface of the rock (Wagner and Leach, 1959; Johnson and Dettre, 1969).

2.2.2 Other Methods

Other methods can be used to determine the sands wettability quantitatively or qualitatively. The two main quantitative methods are the Amott Method, based on the fact that the wetting phase is spontaneously imbibed into cores and displaces the non-wetting phase (Amott, 1959) and the USBM wettability index based on the theory that the non-wetting phase needs more energy to displace the non-wetting phase than the one required for opposite displacing (Donaldson et al., 1969).

The most widely applied qualitative method is imbibition. Since it can provide a quick idea about wettability without any special measurement equipment. A core (sample of reservoir rock containing formation water and oil) at irreducible water saturation is submerged into brine and the oil displacement rate from the core is measured. If the rock is strongly water-wet, the brine will be quick imbibed. If no water is imbibed, the rock is oil-wet or neutrally wet. The limitations of this simple method are that other factors, other than wettability, can affect the imbibition of water into the core. For example, relative permeability and viscosity ratio can also affect imbibition rates (time scale for imbibition) and thus, oil-wet and neutrally wet rock may not be distinguished by this method, since no water imbibed may be in both rock (Anderson, 1986b).

Reservoir logs can also be applied to provide indications of wettability (Graham, 1958; Holmes and Tippie, 1977). For example, the idea that the electrical resistivity of an oil-wet rock is higher than the one of a water-wet rock can be applied to determine rock wettability. But all log related methods can only provide a rough indication about rock wettability.

2.3 Wettability Alternation

2.3.1 Crude Oil Effects

Generally, the molecular composition of the heavy crude oil is hard to determine because of the complexity of heavy oil molecules such as resins and asphaltenes. However, SARA fractionation test is a practical method (Woods et al., 2008) to separate heavy oil into four main solubility categories: saturates, aromatics, resins, asphaltenes (SARA). These are not precise components

but rather solubility classes that are applied to individual oils. Therefore, the composition of these fractions can vary significantly from crude oil to crude oil both within the same reservoir and between other reservoirs. Saturates and aromatics are light components of heavy oil; they have minor effects on rock wettability (He et al. 2015). Surface-active agents and interfacially-active asphaltene are mainly included in the asphaltenes and resins SARA categories (He et al., 2015). The molecular structures of typical natural surface-active agents such as naphthenic acid and sulfonic are shown in Figure 2.3. The surface-active compounds are more widespread in resins and asphaltenes of heavy crude oil than in light heavy crude (Anderson, 1986a).

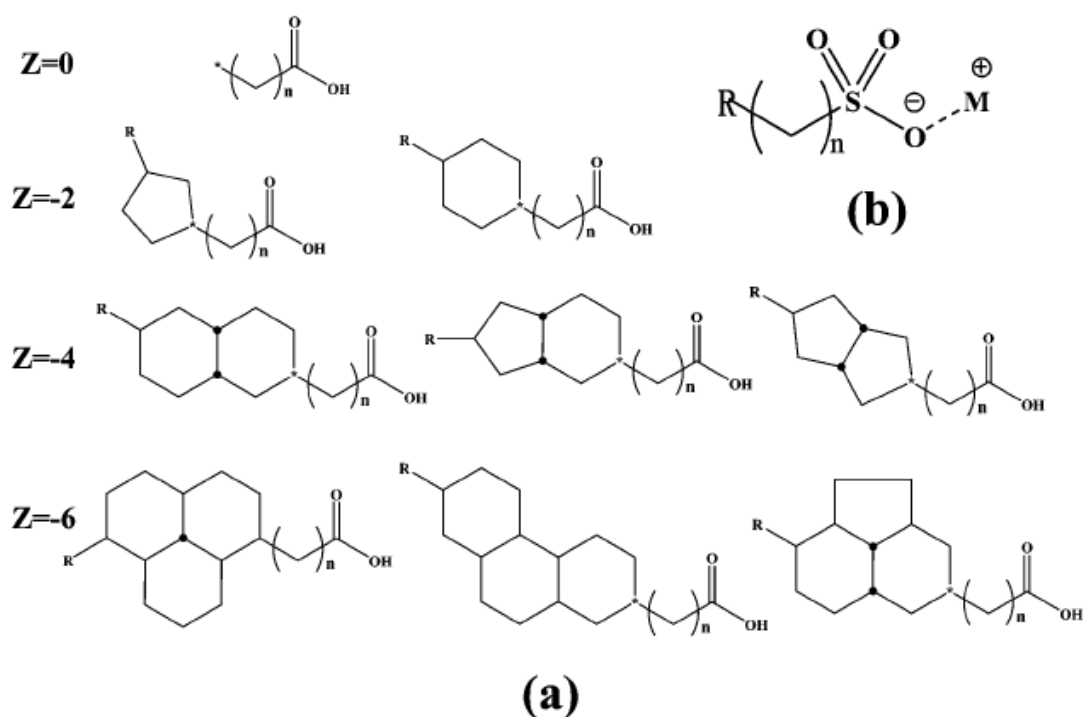


Figure 2.3 Typical structures of naphthenic acids: (a) and sulfonic and (b) R representing the hydrocarbon end (modified from Fan 1991).

Before oil migration into reservoirs, most reservoir minerals are water-wet but these systems may contain non-water-wet mineral such as coal, graphite, sulfur, talc and so on (Anderson, 1986a). After crude oil accumulates in the reservoir, the wettability of reservoir mineral can be altered by the composition of crude oil where polar compounds and depositing organic matter are adsorbed onto the surface of the reservoir rock (Anderson, 1986a; Craig, 1971; He et al., 2015). Polar compounds are the surface-active agents in crude oil. They have both a hydrocarbon end and a polar end. The reason for the alteration of the wettability is that the polar end of asphaltene molecule in naphthenic acid can attach on the surface of rock, and the hydrocarbon is externally exposed, which makes the rock more oil-wet (Anderson, 1986a), as shown in Figure 2.3.

Kumar et al. (2008) reported that asphaltenes can easily be adsorbed on the surface of mica and silica, making the solid surface more oil-wet. Asphaltenes have also been found to become attached to the surface of carbonate minerals. Qi et al. (2013) also found that the high concentration of asphaltene in crude oils can enhance wettability alternation from water-wet to oil-wet rock.

2.3.2 Temperature Effects

Temperature also affects the wettability of reservoir rock but both enhancing and weakening effects of temperature on wettability alteration have been observed. Al-Aulaqi et al. (2011) reported that the wettability of silicate, as measured by contact angle, becomes more oil-wet and a greater amount of asphaltene is adsorbed on the surface of the rock when the temperature is raised. On the other hand, there was no change of the wettability when the silicate was exposed to refined

oil. Nasralla et al. (2011) also found that the contact angle of water on mica sheet submerged in crude oil increased as the temperature was raised. Contrarily, Gupta and Mohanty (2011) observed that the contact angle of brine dropped from 150 to 80° on calcite exposed to crude oil as the temperature was enlarged to 70°C. The different phenomena may be caused by the different composition of solid surface (He et al., 2015).

2.3.3 Organochlorosilanes Treatment

The use of organochlorosilane solutions is a widely applied method to change the wettability of glass and sandstone (clastic) rock from water-wet to non-water-wet. This method can be used to generate both fractional and mixed wettability sands (Fatt and Klikoff Jr 1959; Mohanty, 1983). Generally, organochlorosilane have a non-water-wet organic group and chlorine atoms. The hydroxyl (OH-) group reacts with chlorine on the surface of silicate resulting in exposing the non-water-wet organic group toward the liquid phase (Anderson, 1986a).

2.4 Relative Permeability Measurements

When a single phase flows through porous media, the absolute permeability is obtained from Darcy's law. In the case where another phase is present, the flow of each phase through the porous media is interfered by the other phase. As a consequence, the flow rate of each phase is reduced from the single-phase flow case. Muskat and Meres (1936) developed the relative permeability concept to deal with multiphase flow in porous media. The definition of relative permeability is that it is "a direct measure of the ability of the porous system to conduct one fluid when one or two

fluids are present. These flow properties are the composite effect of pore geometry, wettability, fluid distribution and saturation history” (Craig, 1971). For oil and gas systems, the relative permeability is typically reported as the oil and water relative permeabilities versus the water saturation (the complement of the oil saturation if two phases present) or the oil (or liquid) and gas relative permeabilities versus the liquid saturation. The relative permeability is an important factor control multiphase flow in not only conventional reservoirs (Gates, 2011) but also unconventional reservoirs (Yang et al., 2015; Yang et al., 2016). The relative permeabilities of phases is measured by either steady state or unsteady methods.

2.4.1 Steady State Method

The steady state method for obtaining relative permeability curves is straightforward. Both oil and water in various ratios are injected into the core. When system reaches steady state (meaning the flow rates and pressure drop does not change through time), the pressure difference is measured to determine the relative permeability of each phase. This method is more accurate and has no preferential rock types, since data is measured when the system is steady state. This method usually takes several days to conduct since steady-state flow must be achieved at each set of flow rates.

2.4.2 Unsteady State Method

This method is more rapid to conduct than the steady state method. Johnson et al. (1959) invented the most widely used unsteady state method (referred to as the JBN method) to calculate relative permeability during a water flooding process. The first step of this experiment is restoring initial

reservoir conditions (connate water saturation) by oil injection in a core sample of the reservoir. Then, water is injected into the core sample and the pressure difference and produced fluids are recorded which are subsequently used to calculate relative permeability. Both steady state and unsteady state relative permeability measured on Weiler sandstones (Weiler sandstones are a sandstone originating from Germany) show good agreement (Johnson et al., 1959). A high viscosity oil is usually preferred when using the unsteady state method (Archer and Wong, 1973). Because low viscosity oil is displaced in a piston-like manner, the unsteady method does not provide as accurate relative permeability as the steady state method. A higher injection rate is also required to mitigate inlet and outlet end effects (KYTE and Rapoport, 1958; Craig, 1971).

2.5 Effects of Wettability

2.5.1 Effects on Multiphase Flow in Porous Media

As mentioned above, the wettability of the rock strongly affects multiphase flow and fluid distribution in porous media (Craig, 1971). As shown in Figure 2.4 (a), during water flooding in a strongly water-wet system, water is initially distributed in the small pores and surround the rock grains in the form of a thin water film. Injected water flows within the water films surrounding the grains which pushes oil into the larger porous. In this manner, both oil and water phases form flow paths within the porous matrix. With more water injection, the water flow path is widened whereas the oil path is narrowed and oil in droplet form is left in large pores. After water breakthrough, most of the oil is immobile since it is isolated, in other words entrapped, in the central parts of the larger pores. This results in little oil production from the reservoir after water breakthrough.

Figure 2.4 (b) shows the oil and water flow in an oil-wet system. Initially, oil is distributed in the small pores and wets and surrounds the rock grains. Water flows in the larger pores whereas the oil flows in the smaller pores. When the water saturation increases, the water flow path is raised in the larger pores and the oil flows in the smaller pores. After water breakthrough, little oil is still produced and the water-to-oil ratio steadily increases.

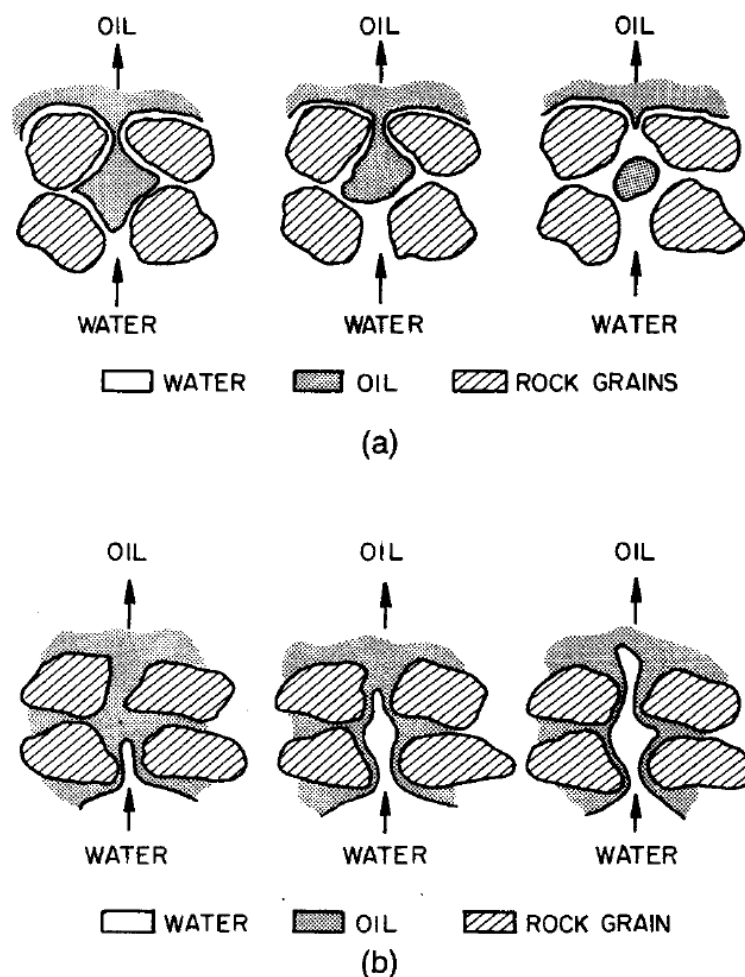


Figure 2.4 Oil and water distribution during a water flooding: (a) strong water-wet rock, (b) strong oil-wet rock. (Raza et al., 1968).

2.5.2 Effects on Relative Permeability

Typical oil-wet relative permeability and water-wet relative permeability curves are shown in Figure 2.5. The relative permeability of water in a water-wet system is lower than that in an oil-wet system. Typically, the water relative permeability in a water-wet system is lower than that which exists in a strongly oil-wet system.

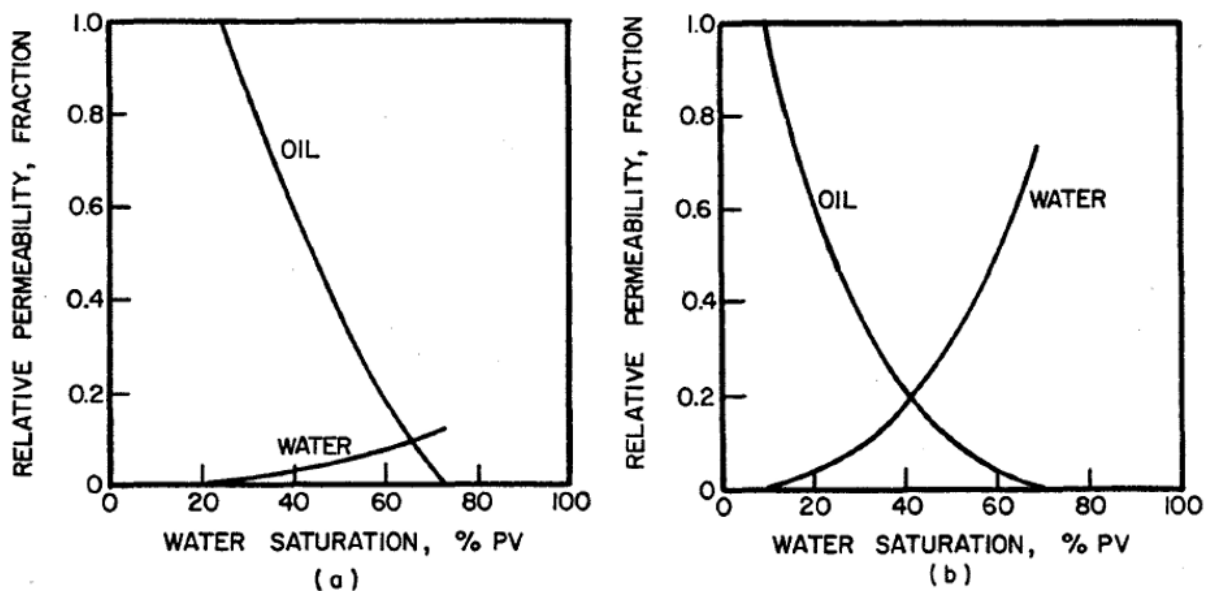


Figure 2.5 (a) Strong water wet relative permeability curves, (b) strong oil wet relative permeability curves (Craig, 1971).

As mentioned above, the wetting phase is preferentially distributed within the smaller pores in the reservoir rock and often exists as a thin film that surrounds the rock grains. Typically, the non-wetting phase is located in the central part of pores. As a result, the non-wetting phase relative

permeability is higher than that of the wetting phase, at a given saturation. At a low non-wetting phase saturation, the wetting phase relative permeability is still not high, because the non-wetting phase preferentially occupies the larger pores. At a low wetting phase saturation, the non-wetting phase relative permeability is high and is close to unity. Craig (1971) also devised three rules to determine the wettability of a reservoir rock based on the water-oil relative permeability curves. For example, in a water-wet system, the irreducible water saturation is typically larger than 20% pore volume. In an oil-wet system, the irreducible water saturation is less than 15% pore volume. Also, if the cross-over point of the oil and water relative permeability curves is greater than 50% (water saturation), the reservoir rock is typically water-wet.

2.6 Wettability of Heavy Oil and Bitumen Systems

Oil sand grains are mainly composed of quartz sand, clay and bitumen. Generally, pure quartz sand and clay are water wet. A large concentration of polar compounds and organic matters in heavy oil and bitumen can potentially change the water-wet grains to oil-wet grains. Thus, the presence of bitumen makes the wettability of oil sands complex.

It is generally accepted that there is a film of water covering on the surface of the oil sand grains (Takamura, 1982; Czarnecki et al., 2005). Figure 2.6 shows a conceptual diagram of the water film between sand grains and bitumen. Two scenarios are postulated to explain the formation of water film. The first scenario states that water is initially filled the porous medium and then oil migrated into the large pores. As a result, the oil occupies the central parts of the pores and a water film is adjacent to the grains. The second scenario suggests that the sand is initially dried without any

fluid saturation. First, oil migrated into these pores and then water invaded into the small pores under the action of capillary pressure.

Due to the presence of the water films, the bitumen is separated from the sand grains, and cannot alter the wettability of the sands by direct adsorption of polar components or organic matters from the oil phase. As a result, the Athabasca oil sands is considered water-wet. The water films are also a significant feature of the Athabasca oil sands which enables steam-based recovery processes (Czarnecki et al., 2005).

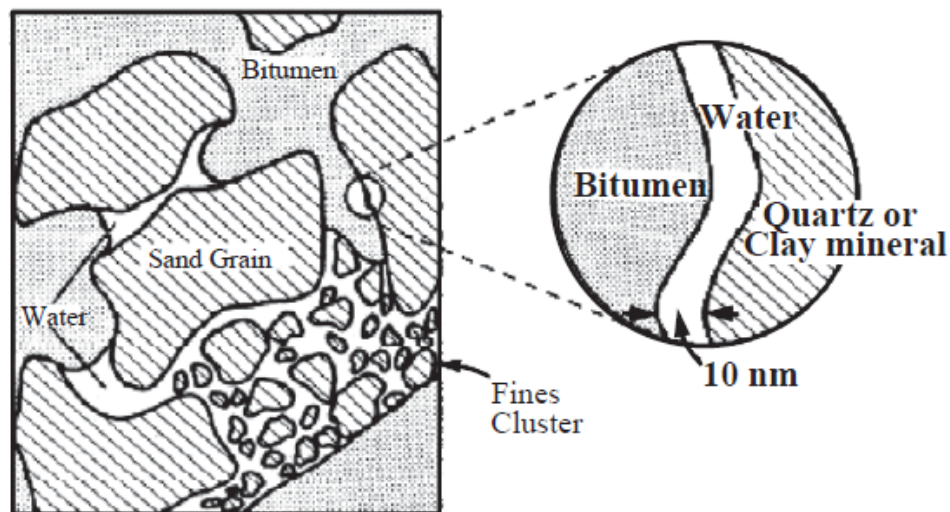


Figure 2.6. The structure model of Athabasca oil sands shows the presence of water film between bitumen and sand grains (Takamura, 1982).

The stability of the water film directly affects the wettability of oil sands. Czarnecki et al. (2005) analyzed the stability of water films. When thickness of water film is above about 50 nm, it is

referred as a thick water film. The balance between capillary and gravity forces controls the stability of a thick film, and in general, it is stable. If the water film thickness is less than 50 nm, the main control forces are electrical double layer forces (stabilizing the water film on the surface of grain) and London-van der Waals forces (destabilizing the film) (Czarnecki et al., 2005). The literature shows that an integrated effects of pH value, film thickness and particle limiting radius control the stability of water film, as shown in Figure 2.7. The unstable areas are above curves with different brine pH values, and the stable areas are below these curves.

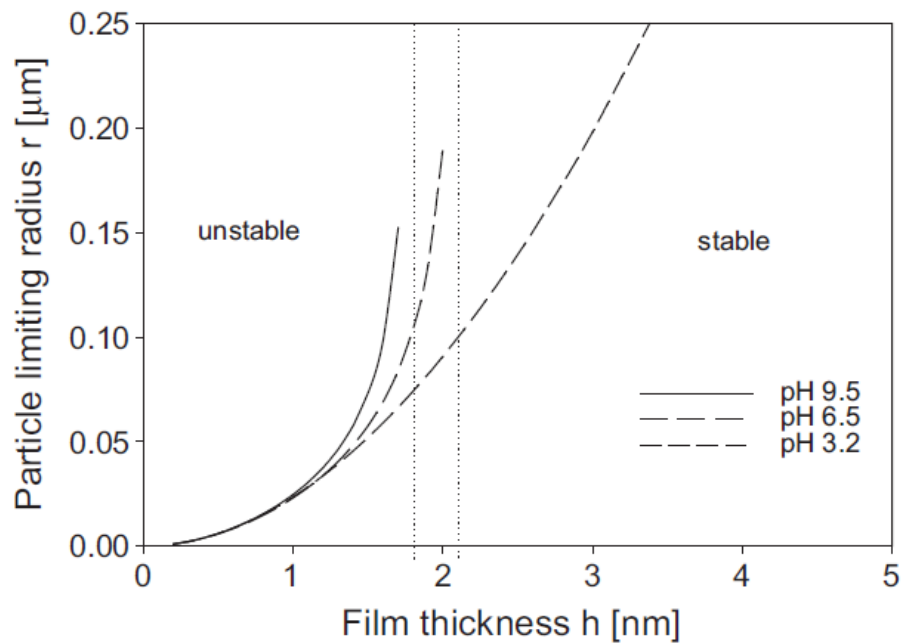


Figure 2.7 The unstable areas of water film are above curves with different brine pH values, and the stable areas of water film are below these curves (Czarnecki et al., 2005).

Zajic et al. (1987) examined the nature of water films around Athabasca oil sand grains. They firstly freeze-fractured test samples, then examined the water and bitumen phase distribution by using transmission electron microscopy (TEM). The water was preserved in droplets mixed with bitumen, and was found to be only distributed in the large pores among grains. No water film was observed from the TEM images. Their observations suggest that the oil sand will be oil-wet due to exposure to bitumen. However, the process of freezing the samples likely led to shrinkage of the water films into discrete water droplets and thus the observations may have been in error.

In terms of heavy oil and bitumen systems, the high content of asphaltene strongly affects the wettability of rock directly exposed to heavy oil and bitumen. Dubey and Waxman (1991) reported that the adsorption of asphaltene on grains alter the water-wet sand into weak water-wet or neutral wet sand. The retained asphaltene after desorption is also calculated in their work. In their study, a core with adsorbed asphaltene was dried at room temperature. They did not test the effects of a high temperature drying process on asphaltene adsorption/desorption. A similar wettability alternation was conducted by Sharma and Wunderlich (1987) to make mixed wet-cores by asphaltene precipitation.

2.7 Relative Permeability Measurements in Heavy Oil and Bitumen Systems

Relative permeability directly determines multiphase phase flow in porous media. In terms of heavy oil and bitumen systems, most relevant research has focused on the effects of temperature on relative permeability and the results are not all consistent.

Miller and Ramey Jr. (1985) evaluated relative permeability of consolidated and unconsolidated sandstones at different temperatures by using the unsteady state method. Figure 2.8 displays that the relative permeability at elevated temperature is almost the same. They concluded that an increase of the temperature does not change the residual saturation and relative permeability, and that viscosity instabilities, capillary end effect, and difficulties in maintaining system material balance affected the relative permeability rather than that of temperature.

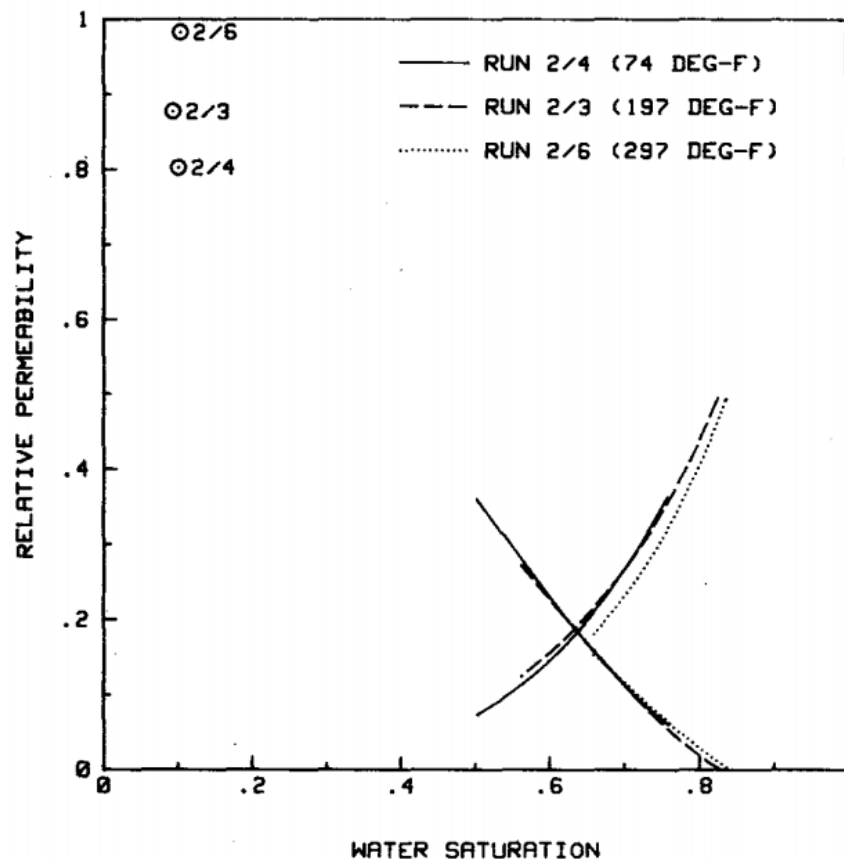


Figure 2.8 Relative permeability at different temperatures (Miller and Ramey Jr, 1985).

Polikar et al. (1990) investigated the relative permeability of the Athabasca oil sands at high temperature conditions. They used the high-grade oil from Athabasca oil sand and deionized water as testing fluid and applied both steady state and unsteady state methods to evaluate the relative permeability of oil and water phases. Figure 2.9 displays the measured relative permeability curves by both methods at temperature of 125°C. They found temperature has no obvious effects on relative permeability. The observed variation of end points may be caused by sand pack heterogeneity. Both results of steady state and unsteady state methods are coincidence, which represent the similar displacement process during both experiment.

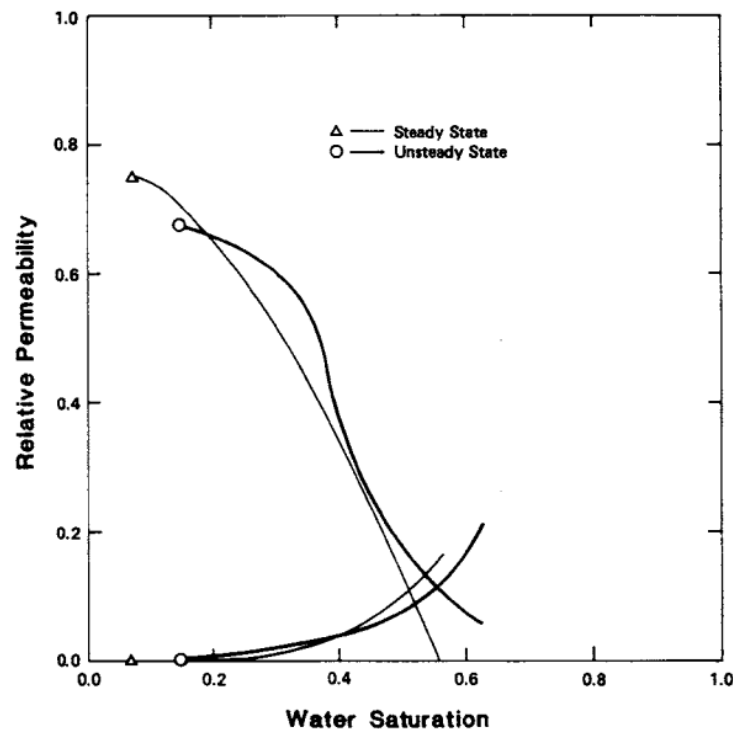


Figure 2.9 The relative permeability measured by steady and unsteady state method at the temperature of 125°C (Polikar et al., 1990).

Another main contribution reported by Polikar et al. (1990) was a comparison of the relative permeability of oil and water in Athabasca oil sands measured by different experiments as shown in Figure 2.10. The oil relative permeability by Polikar et al., 1990 is convex, whereas the ones measured by other researchers exhibited a concave shape. Based on these differences, they thought that it is necessary that a wide range of relative permeability should be applied for different recovery processes for Athabasca oil sands.

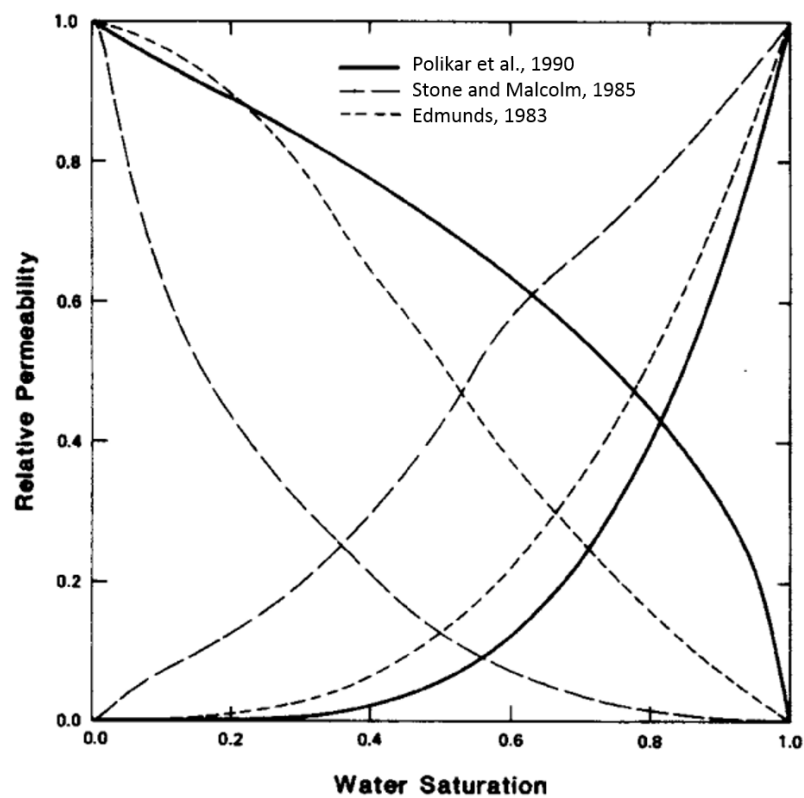


Figure 2.10 Normalized relative permeability measured by Polikar et al., 1990, Stone and Malcolm, 1985 and Edmunds, 1983.

Schembre et al. (2006) stated that for Athabasca systems, the wettability of the rock became more water-wet as the temperature was raised. They used X-ray computerized tomography (CT) to determine the water saturation during imbibition experiment at high reservoir temperatures, and then the relative permeability and capillary pressure were evaluated from the observed saturation data. Figure 2.11 shows that as temperature is raised from 120°C to 180°C, the maximum water relative permeability dropped and the oil relative permeability generally increased at a given water saturation, indicating that the sand became more water-wet.

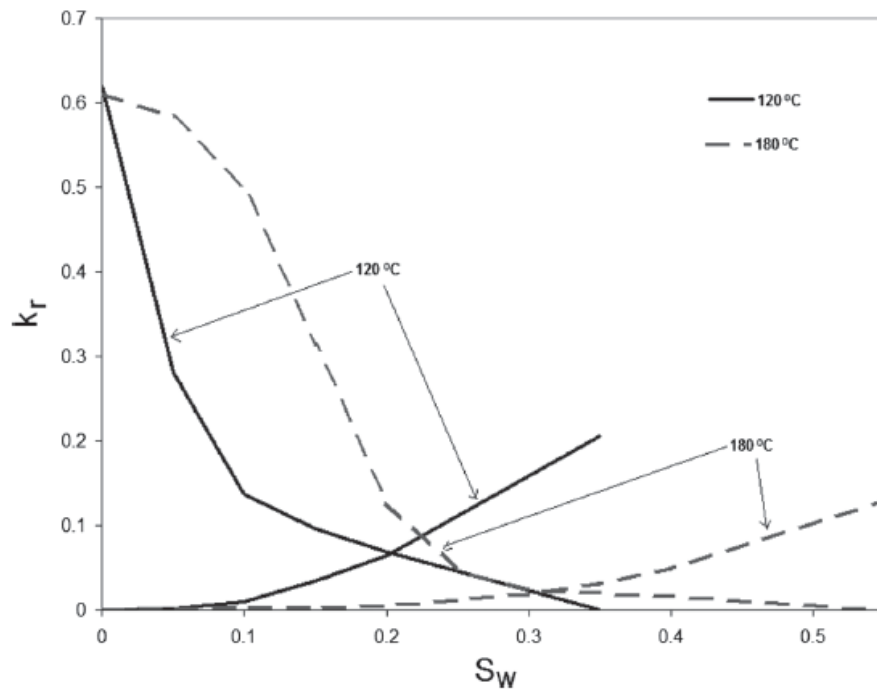


Figure 2.11 Relative permeability of brine and crude at 120°C and 180°C (Schembre et al., 2006).

Bennion et al. (2006) investigated lab-measured relative permeability of western Canadian heavy mostly McMurray sandstones, were tested. Different original crude oils with viscosities ranging from 8,000 cP to 1,000,000 cP, was used. They mainly applied the unsteady state method to calculate relative permeability. The relative permeabilities were grouped into the low temperature range (less than 80°C), shown in Figure 2.12, and high temperature range (more than 150°C), displayed in Figure 2.13. They also derived correlations between water saturation and relative permeability under both low temperature and high temperature conditions. The results demonstrate that the relative permeability curves are dependent on temperature.

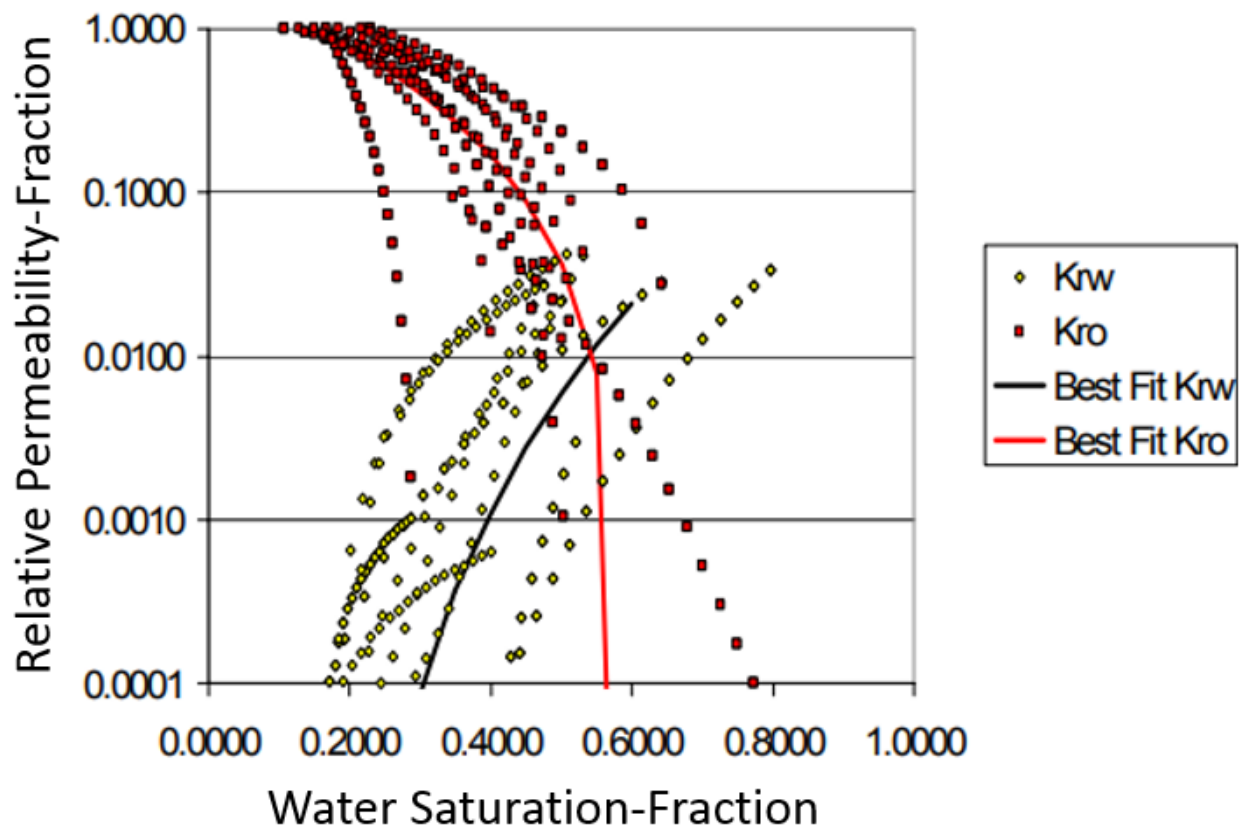


Figure 2.12 Relative permeability summary of heavy oil systems under low temperature (less than 80°C) (Bennion et al. 2006).

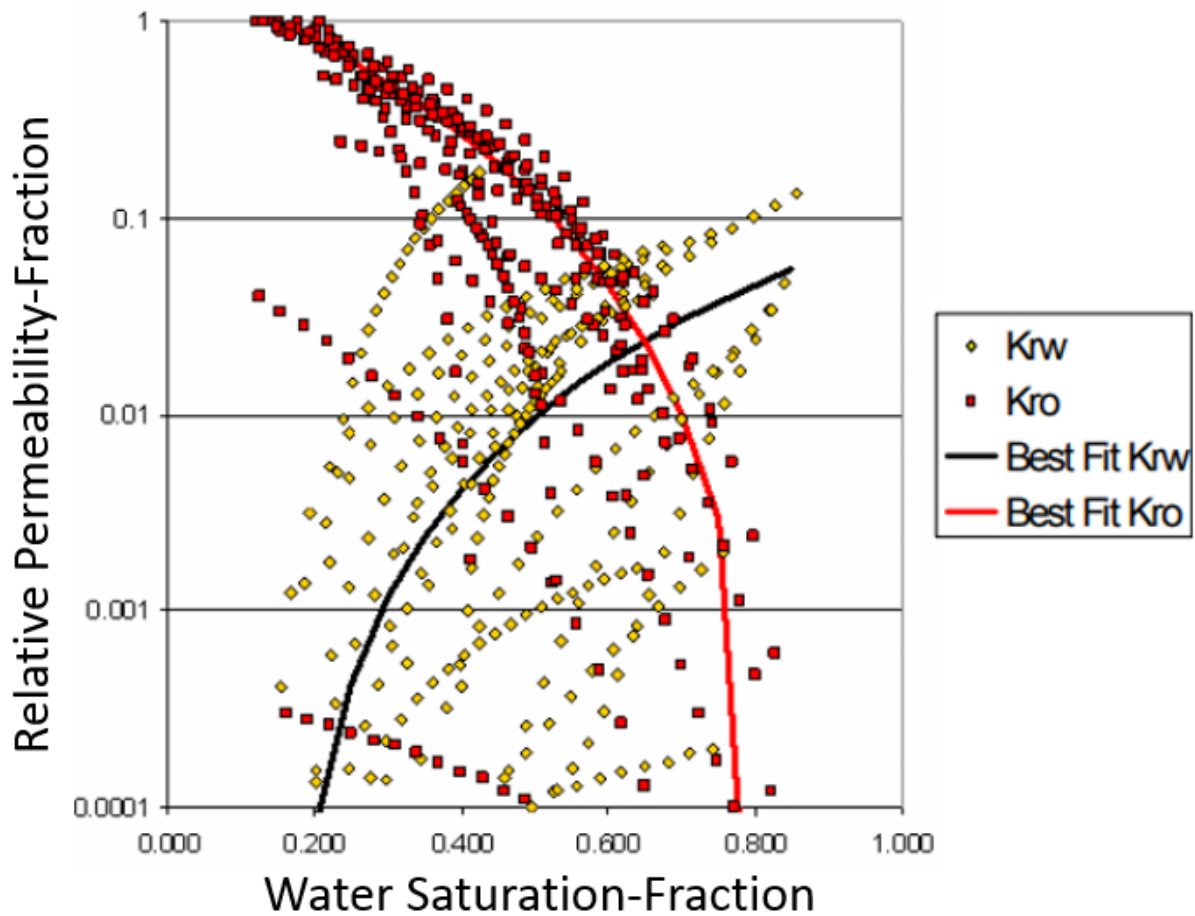


Figure 2.13 Relative permeability summary of heavy oil systems under high temperature (more than 150°C) (Bennion et al. 2006).

2.8 What is missing in the literature?

Generally, there are a few relative permeability curves for oil sand systems in the literature. However, even only for Athabasca oil sands, relative permeability curves estimated from different research studies are significantly different. Many factors affect relative permeability of oil sands systems such as wettability, viscosity ratio, temperature, lithology heterogeneity and even recovery

processes. Although some work has focused on wettability of oil sands systems, but there is a lack of further discussion about wettability effects on relative permeability in oil sands systems. Therefore, a determination of wettability of an oil sand system and an estimation of relative permeability under a given wettability and other conditions are still very crucial for the design of oil sand deposit development. This is the subject of this thesis.

Chapter Three: Experimental Apparatus and Measurements

In this Chapter, the experimental apparatus is described.

3.1 Tube Flow Measurement Device

Figure 3.1 and 3.2 display the schematic diagram and image of water flooding equipment, respectively. The sand pack is a plastic cylinder tube of 32 cm length and 5 cm diameter packed with prepared sand, displayed in Figure 3.2. The experimental system consists of a core holder, high pressure pumps, differential pressure gauge, flow lines, control valves, and a volume measurement system.

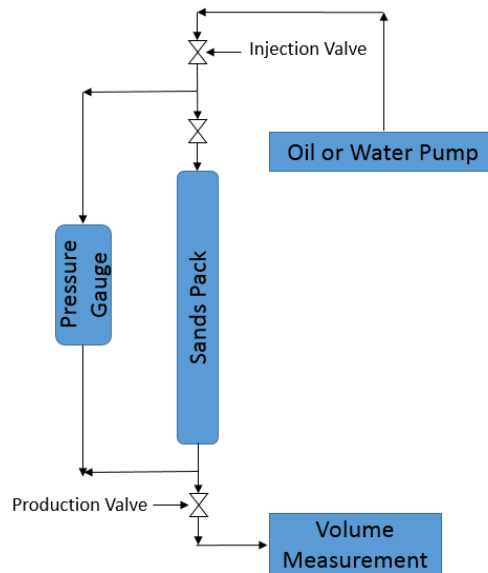


Figure 3.1 Schematic diagram of water flooding equipment.



Figure 3.2 Plastic sand pack holder.

The pressure gauge (Model TJE, Honeywell) is attached in the inlet and outlet of the sand pack, and the pressure difference is automatically recorded by the data acquisition system, shown in Figure 3.3. The data acquisition system uses LabviewTM (NI, 2017) for automated data recording. Both a high-pressure water pump (SYRINGE PUMB Model 500D, TELEDYNE ISCO), illustrated in Figure 3.4, and a high pressure oil pump (PUMP Model 307, GILSON), shown in Figure 3.5 were used in the experimental apparatus.

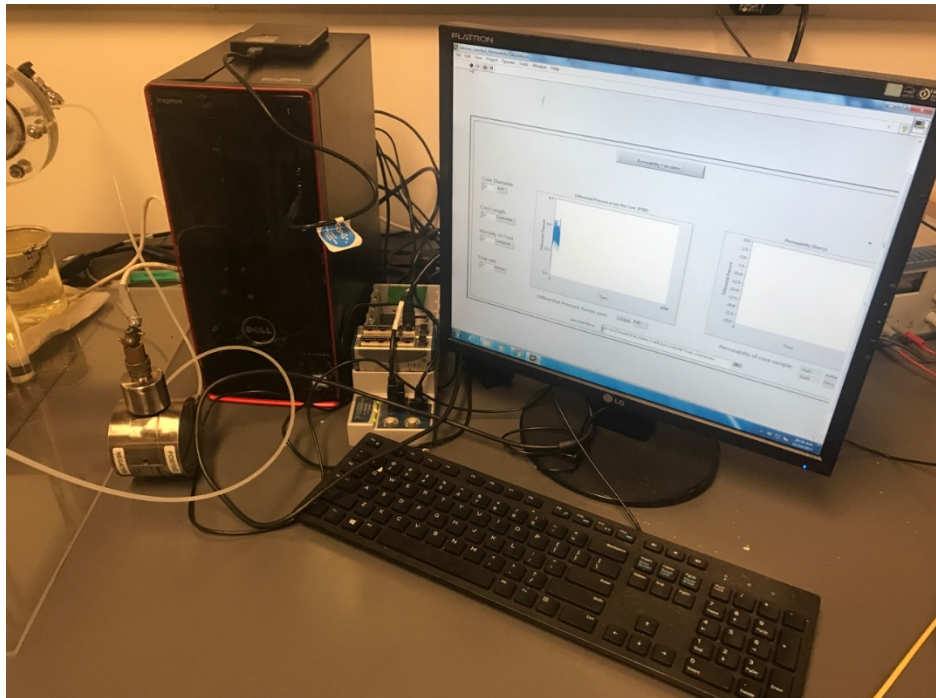


Figure 3.3 Pressure gauge and recording computer.



Figure 3.4 Water pump.



Figure 3.5 Oil pump.

3.2 Sand Grain Diameter

The sand grains used in the research reported in this thesis were obtained from McMurray Formation oil sands obtained from an operating oil sands mine in Ft. McMurray, Alberta. To obtain the grain size distribution, an ANALYSETTE 28 Image Sizer, FRITSCH was used. The area equivalent grain diameter data is presented in Figure 3.6. The data reveals that the average size of the sand grains is equal to about 162 microns.

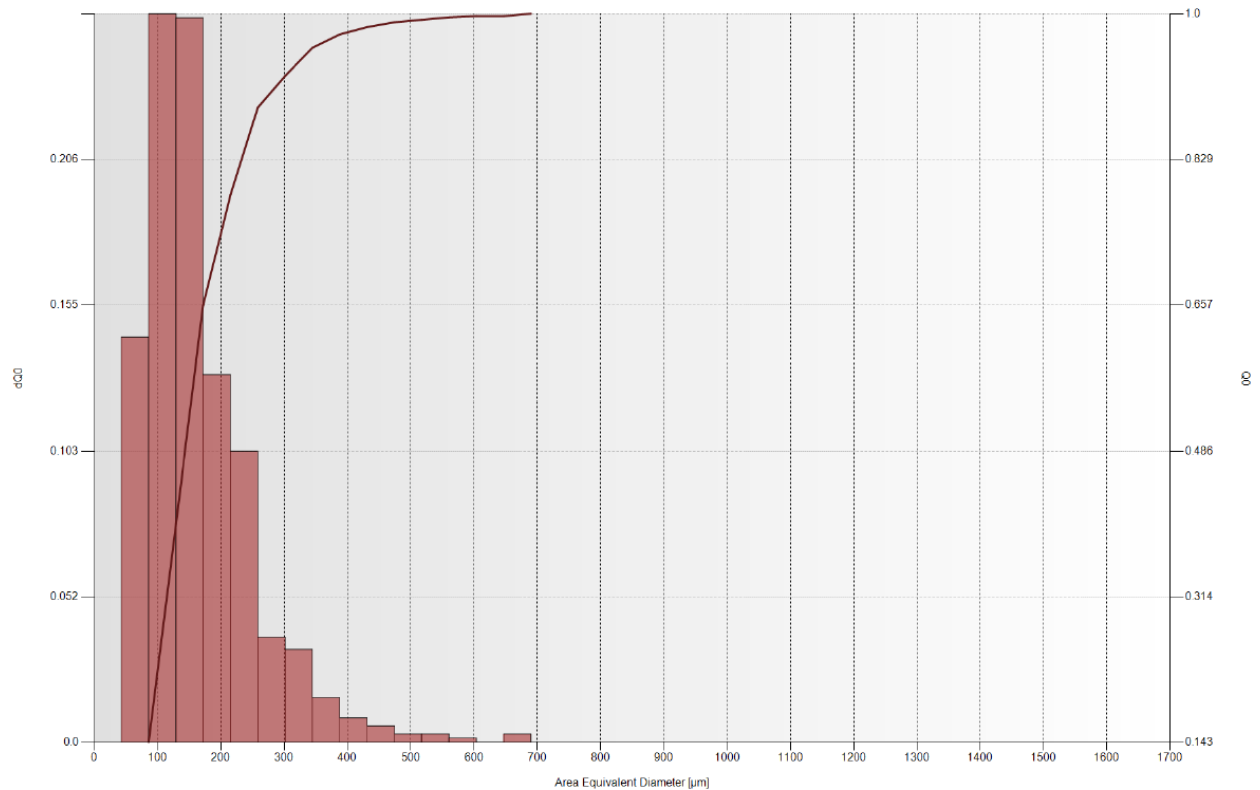


Figure 3.6 Area equivalent sand grain diameter analysis results.

3.3 Sand Density

The most abundant component in the sands is quartz which has a mass density equal to 2.648 g/cm³. To evaluate the density of the sand grains used in the experiments conducted in the research documented in this thesis, the oil sands were initially cleaned of bitumen. This was done by soaking the oil sand in toluene multiple times. After the oil and toluene was removed from the sand, it was weighed. The volume of the sand was then measured by placing it into a test tube filled with water and observing the change of the level of the water. This procedure was done in

triplicate and the data is reported in Table 3.1. The average density determined is equal to 2.651 g/cm³. This is close to the value for pure quartz.

Table 3.1. Sands density measurement result.

Sand Weight (g)	Sand Volume (cm ³)	Density (g/cm ³)	Average (g/cm ³)
11.24	4.25	2.645	2.651
14.06	5.30	2.653	
17.27	6.50	2.657	

3.4 Sand Pack Bulk Volume Measurement

The bulk volume of the sand pack was determined from the inner diameter and length of the sand pack holder. The volume calculated from the dimensions is equal to 640 ml. The sand grain volume, calculated by the amount of sand used to fill the pack and the grain density, is equal to 388 ml. The pore volume is then equal to 640 ml – 388 ml = 252 ml. The porosity of the sand in the pack is then equal to 252/640 = 0.394.

3.5 Fluids

In the experiments reported in this thesis, deionized water was used. The oil used was paraffin oil, coloured white (Anachemia, 66102-540).

3.6 Fluid Viscosity

The viscosity of the oil versus temperature was measured by using a Brookfield rheometer (Stabinger Viscometer 3000, Anton Paar). The oil viscosity was measured at three different temperatures and is plotted in Figure 3.6. The viscosity of the water used in the experiments was found equal to 1 cP. All experiments were conducted at room temperature (21°C); at this temperature, the viscosity of the oil was equal to 80 cP.

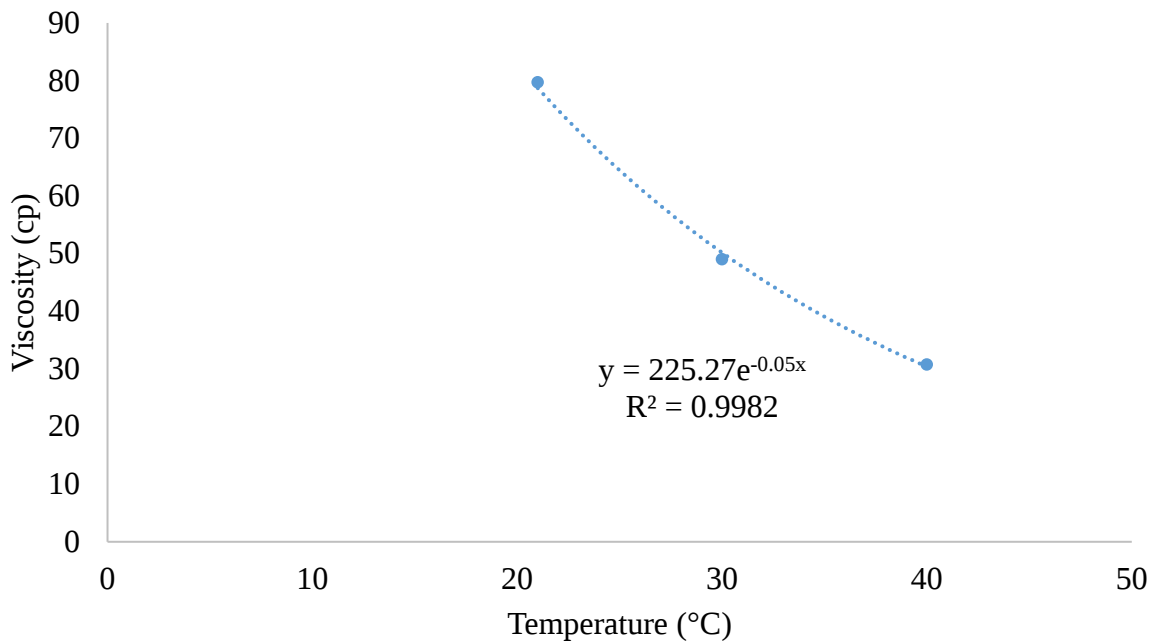


Figure 3.6 Measured oil viscosity under different temperatures.

3.7 Interfacial Tension

The interfacial tension between the mineral oil and water system is 40.03 mN/m, measured by Du Nouy ring method (Model TensioCAD-M, CAD Instruments). This was determined as the average of three experiments.

Chapter Four: Preparation of Water-Wet and Oil-Wet Sands

This chapter describes a new method to prepare oil-wet and water-wet sands. The method starts with McMurray Formation oil sand – this is a quartz rich sand that has grains, mostly between 100 and 200 microns in size. For the sands used here, the average grain size is equal to 162 microns.

Sand from McMurray Formation oil sands from a mine is used in the research documented in this thesis. As shown in Figure 4.1, the original unconsolidated oil sands are surrounded and saturated with bitumen.



Figure 4.1 Original McMurray Formation oil sands obtained from an oil sands mine in the Ft. McMurray area.

The bitumen and its high viscosity hinder sands wettability measurements and the efficiency of proposed water injection, so the measured sands are firstly cleaned to remove bitumen to get suitable sands for experiments.

4.1 Preparation of Water-Wet Sands

Based on API's recommendation (API, 1960), toluene can be used to clean asphaltic crudes and in the research documented in this thesis, toluene is applied as the cleaning solvent. First, the oil sand is mixed with toluene with a volume ratio of toluene-to-oil sands equal to 2:1. Second, after the bitumen is totally dissolved in the toluene, the liquid is passed through a filter and the solid is collected. This procedure is repeated multiple times (typically three to four times) until the filtrate is completely clear (transparent) with no bitumen stain.

A single solvent is not effective for completely cleaning the bitumen from the oil sands (Gant and Anderson, 1988). Even though the filtrate is transparent, some residual bitumen and toluene remain within the sand matrix. In the subsequent step, the sand matrix is washed with water and as a consequence, the remaining toluene and bitumen is separated from the sand and a filter is used to collect the filtrate of toluene/bitumen drops and water. This step is repeated again until no more oil drops can be observed in the liquid filtrate. In the next step, the cleaned sands are then dried in an oven at 130 °C for 24 hours. An example of the cleaned sands is shown in Figure 4.2. The sand obtained from this procedure is water-wet (described below).



Figure 4.2 Cleaned water-wet sands.

4.2 Preparation of Oil-Wet Sands

To obtain the oil-wet sand, a different cleaning procedure is used to alter the wettability of the sand. In the first step, toluene is still used to dissolve bitumen but the volume ratio of toluene-to-oil sand is 1:1. After the sand is totally mixed with toluene, the liquid is filtered. As with the previous procedure, some of the toluene and residual bitumen remains in the sand matrix. Since toluene has dissolved all the bitumen into an oil phase, it covers the surfaces of the sand grains. These sands are then put into the oven and set the oven temperature at 130°C to evaporate the toluene and any light ends that were originally in the oil sand. After sands are totally dried, the residual bitumen is evenly coated on the sand grains. In this case, the sand matrix is brown in color as shown in Figure 4.3.



Figure 4.3 Oil-wet sands after the first time washing and drying.

The dried sands are further repeatedly cleaned by toluene again until the mixed liquid is transparent after which the sand is washed with water as was done in the preparation of water-wet sands. The final cleaned oil-wet sands have a grey color as shown in Figure 4.4.



Figure 4.4 Cleaned oil-wet sands.

4.3 Water Contact Angles

Many different methods have been proposed to measure the wettability of sands. In this thesis, the contact angle method is applied for quantitative wettability measurements. As shown in Figure 4.5, the sands are attached onto the bottom surface of the fluid container by using a two-sided adhesive tape. Next, the container is fully filled with mineral oil and then a 1 ml drop of water is placed on the surface of sands. The droplets are then imaged from the side to obtain a cross-sectional view of the droplet of water on the sand grains.

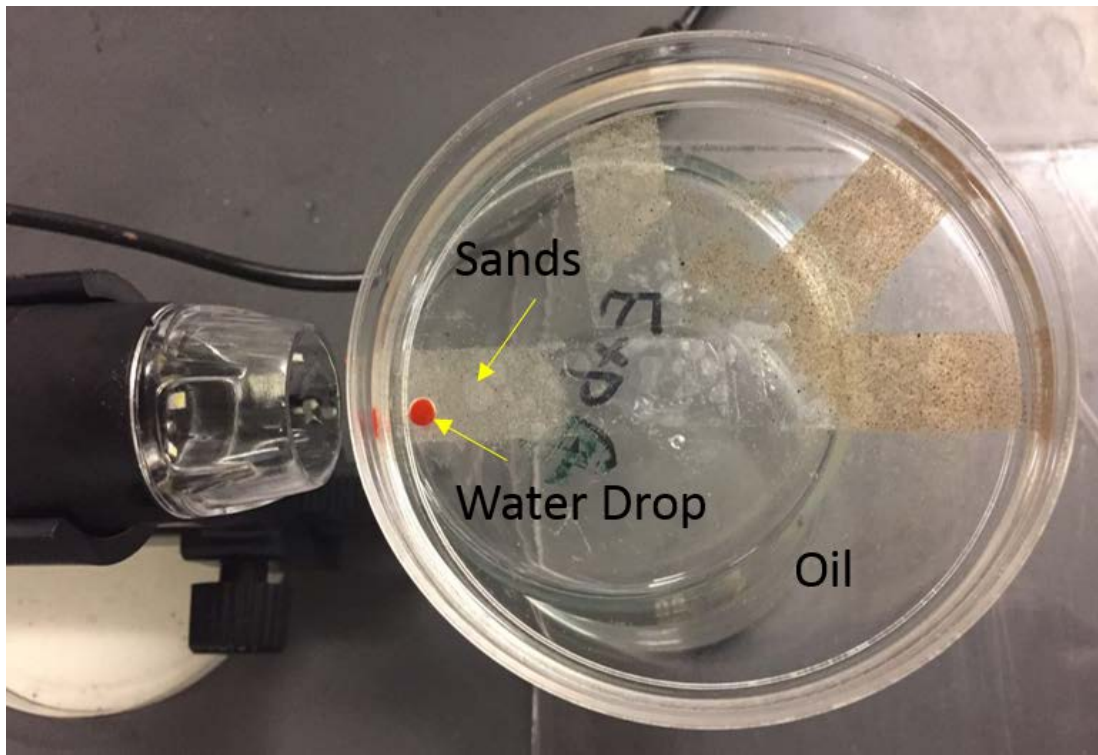


Figure 4.5 Contact angle measurement device.

Ten contact angles of water-wet sands have been measured, and range from 40° to 60° . Figure 4.6 displays the water-oil droplets on the water-wet sands for two experiments; the contact angles are 42° and 56° , respectively. Ten contact angles of oil-wet sands have been measured, and range from 130° to 160° . Figure 4.7 shows the water-oil contact angles on oil-wet sands for two experiments. The contact angles are 158° and 135° , respectively.

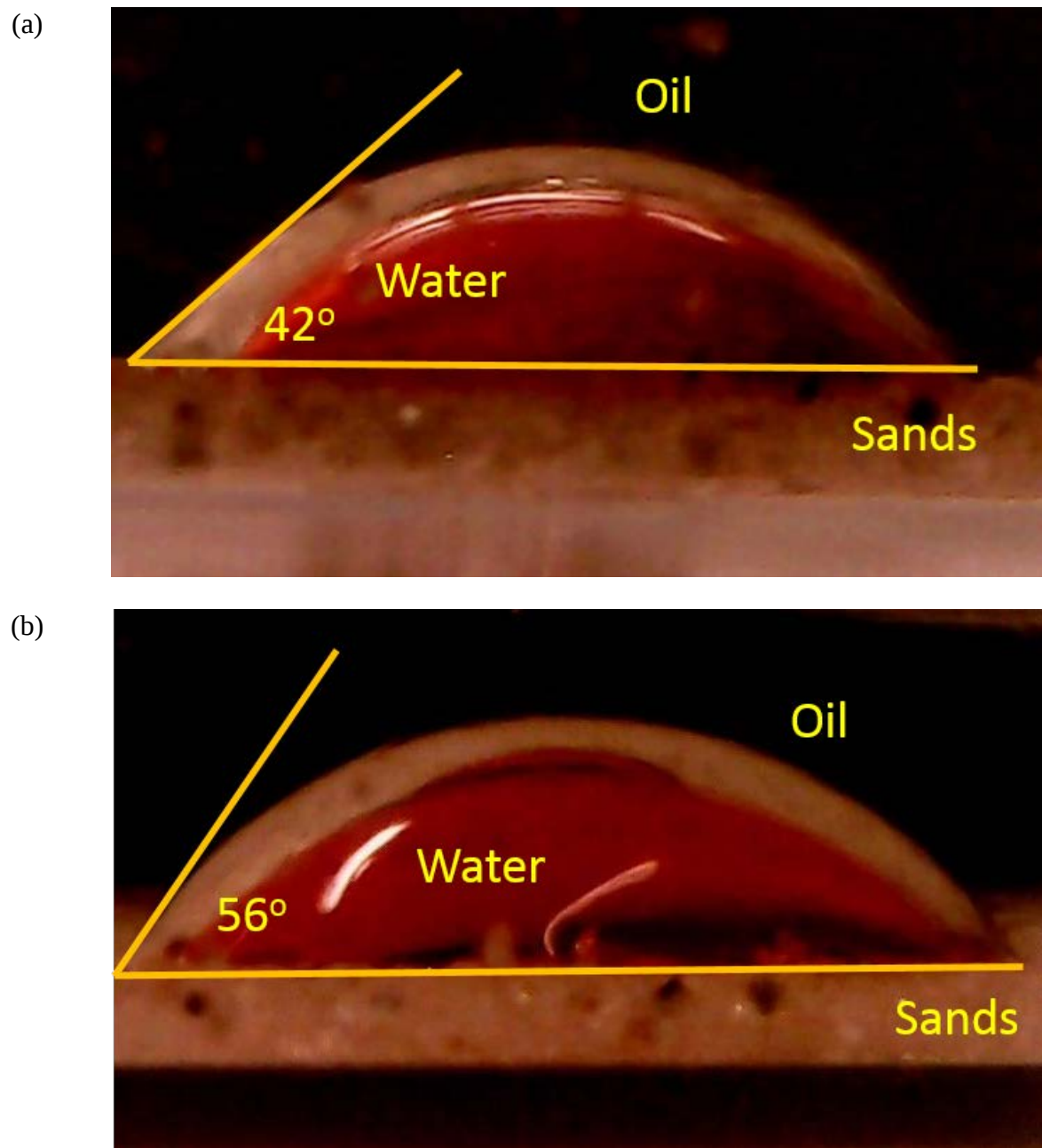
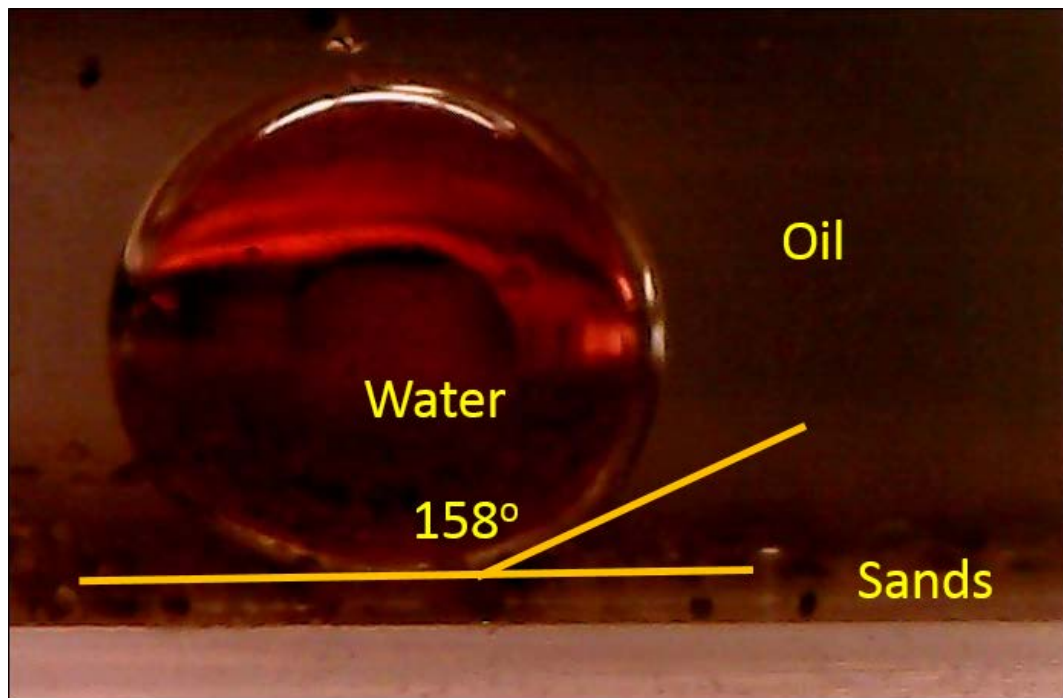


Figure 4.6 Two measured water-oil contact angles on water-wet sands.

(a)



(b)

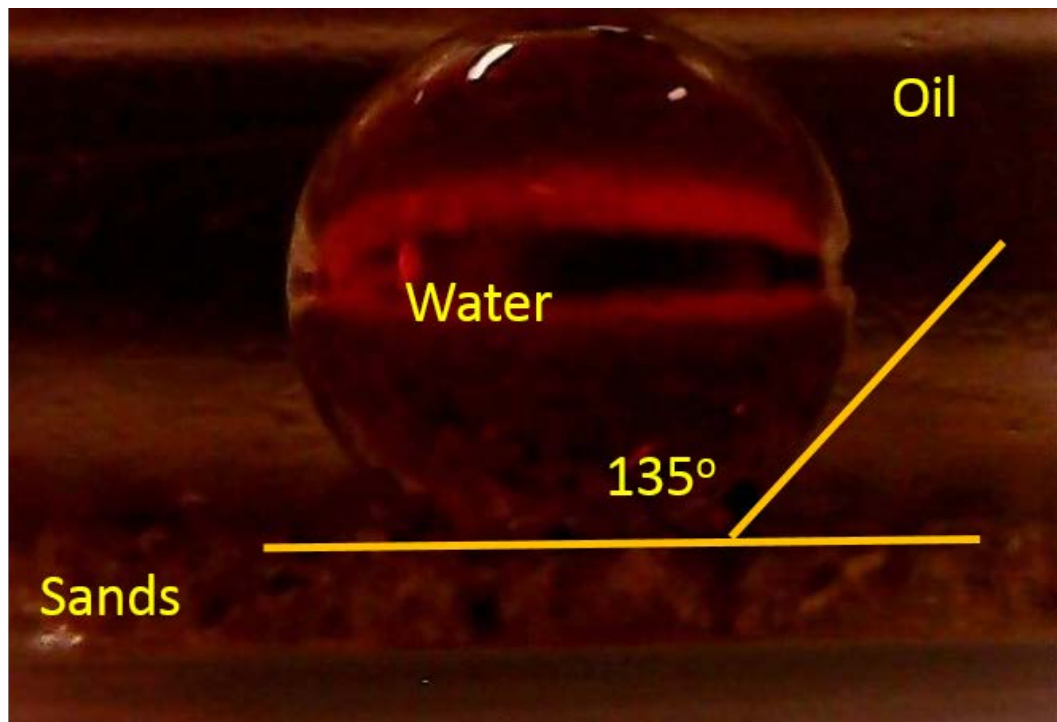


Figure 4.7 Two measured water-oil contact angles on oil-wet sands.

Chapter Five: Water Flooding Tests of Water-Wet and Oil-Wet Sands

This chapter reports on experiments on water flooding into both water-wet and oil-wet sand. First, the absolute permeability of the sand pack is determined by using water injection into the pack under single phase flow. Thereafter, the sand pack is filled with mineral oil after which water flooding is conducted.

5.1 Water-wet Sand: Absolute Permeability Measurements by Water Injection

In this step, water is injected into the sand pack at a set of constant flow rates and the pressure difference is measured. To ensure that an accurate estimate of the absolute permeability is determined, the water is injected at multiple flow rates. Five experimental tests were conducted. In each test, the water injection flow rate ranges from 5 ml/min to 80 ml/min. When a flow rate is changed during a test, the pressure difference is measured only when the system reaches steady state (pressure difference is constant versus time). Then, the Darcy equation is applied to calculate the absolute permeability.

In total, the absolute permeability was estimated from 55 different experimental runs on the same sand pack. The average value determined from the experiments, each one analyzed by using Darcy's law, is equal to 6.9 Darcy. Because the sands are unconsolidated, the structure of porous media can be modified under different fluid pressure and thus, there is variability of the calculated permeability. Therefore, a simple average permeability is not sufficient to represent the real

absolute permeability. An alternative is to plot the flow rate versus pressure difference and then regress the data to determine the absolute permeability. The data is plotted in Figure 5.1. A linear regression of the data yields an absolute permeability equal to 7.3 D with a correlation coefficient (r^2) equal to 0.94.

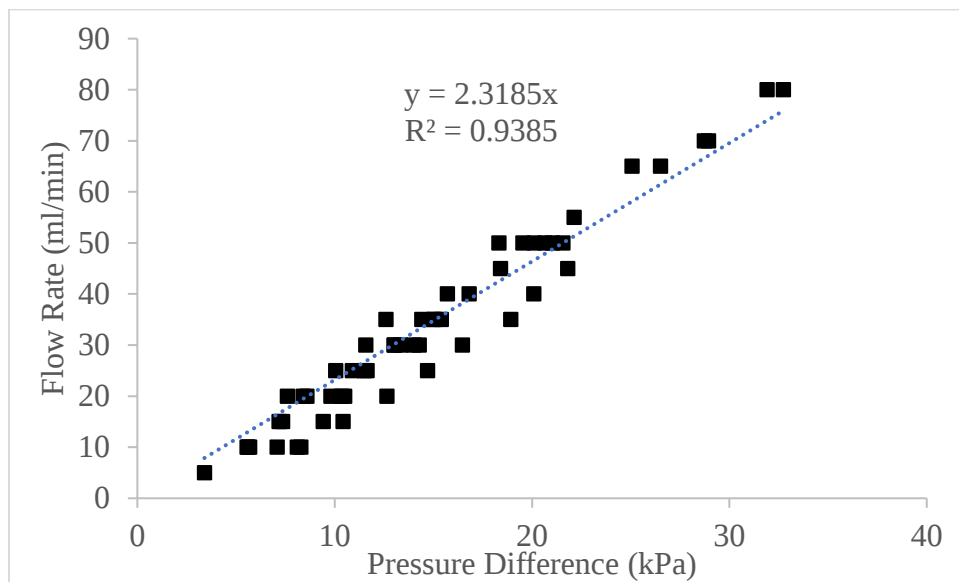


Figure 5.1 Relationships between flow rates and pressure differences during absolute permeability measurements of water-wet sands.

5.2 Water-wet Sand: Modelling Reservoir Conditions by Mineral Oil Injection

Mineral oil is injected into the water-wet sands fully saturated with water to establish the initial conditions of the oil-filled sand pack. The oil is injected into the sand pack at 2.5 ml/min. This is continued until the out flow contains only oil. Figure 5.2 shows out flow volumes of water and oil. The breakthrough time can be observed at the 87 minutes. After breakthrough, the water volume

increases slightly and the water fraction is lower than 2%. After 120 minutes of oil injection, no water is produced.

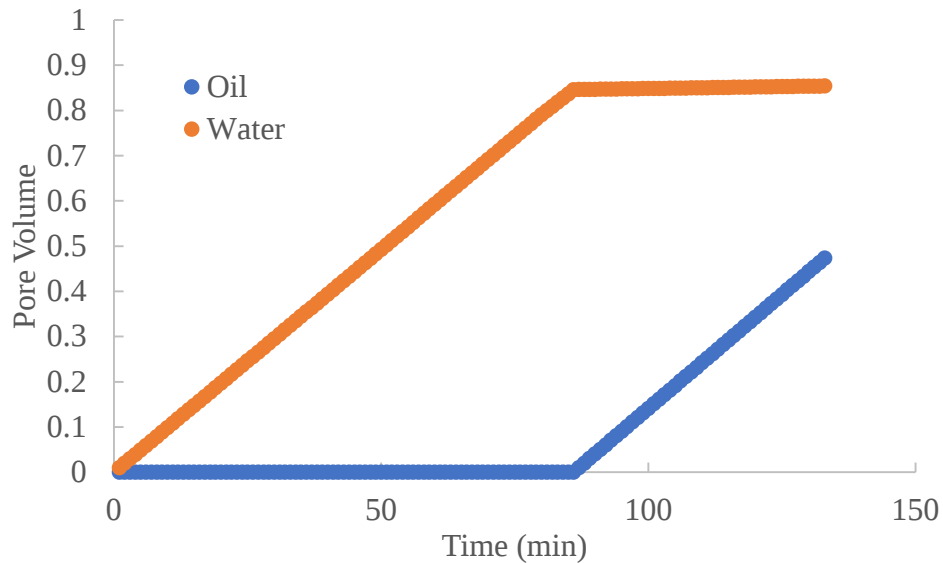


Figure 5.2 Flow-out pore volumes of water and oil during the first oil injection in water-wet sands.

Figure 5.3 displays the relationship between the pore volumes of injected oil and pressure difference. The pressure difference builds up as the oil is injected into the sand pack. This is because the increasing saturation of the relatively high viscosity oil results in a higher pressure requirement to maintain a constant oil injection rate. A bigger jump occurs at the time of oil breakthrough, since an oil flow path is formed and smaller amounts of water flow out from the pack. Beyond this point, the pressure difference increases slowly due to the very small additional oil added to the pack after breakthrough.

By accounting for the mass balance of oil and water around the core during oil injection, the irreducible water saturation is found to be equal to 12%. The oil effective permeability at the irreducible water saturation, calculated by using the Darcy equation (since the system is essentially at steady state), is found to be equal to 7 Darcy. The pressure jump at the breakthrough point may be caused by capillary pressure.

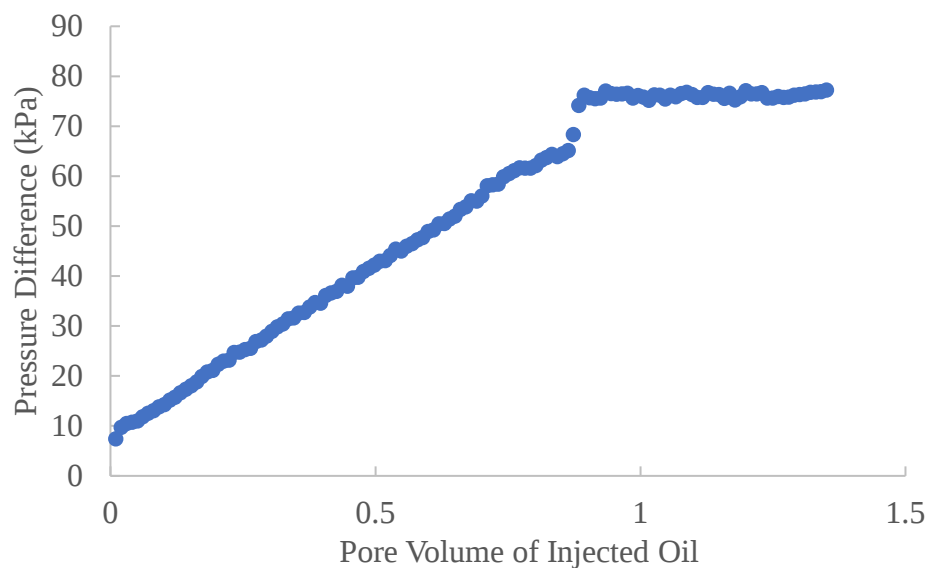


Figure 5.3 Pressure difference versus injected oil pore volume during the first oil injection in water-wet sands.

5.3 Water-wet Sand: Water Flooding

In this experiment, a constant water flow rate 5.5 ml/min is injected into the sand pack. Injection is stopped when the production water cut is greater than 99.5%. Figure 5.4 shows the produced volumes of water and oil from the sand pack. Water breakthrough occurs at 14 minutes. After breakthrough, the oil cut gradually drops from 100 to 0.5%, as shown in Figure 5.5. Figure 5.6

displays that the pressure difference severely decreases before the water breakthrough point and then after breakthrough, declines at a slower rate. The main reason is that the pressure difference falls is due to the reduction of the oil saturation. Before breakthrough, both water and oil are displaced by the injected water. After breakthrough, a continuous water flow path is formed through the porous pack and beyond this point, water is preferentially moved through the water path although oil is still displaced, at lower rate, from the sand pack. The oil recovery factor at the time of water breakthrough is equal to 32%; at the end of the flood, the ultimate oil recovery factor is 55%. When pressure difference is steady and almost no oil can be displaced, the system is in a steady state. The residual oil saturation calculated by mass balance is 0.41, and the corresponding k_{rw} based on Darcy equation is 0.23.

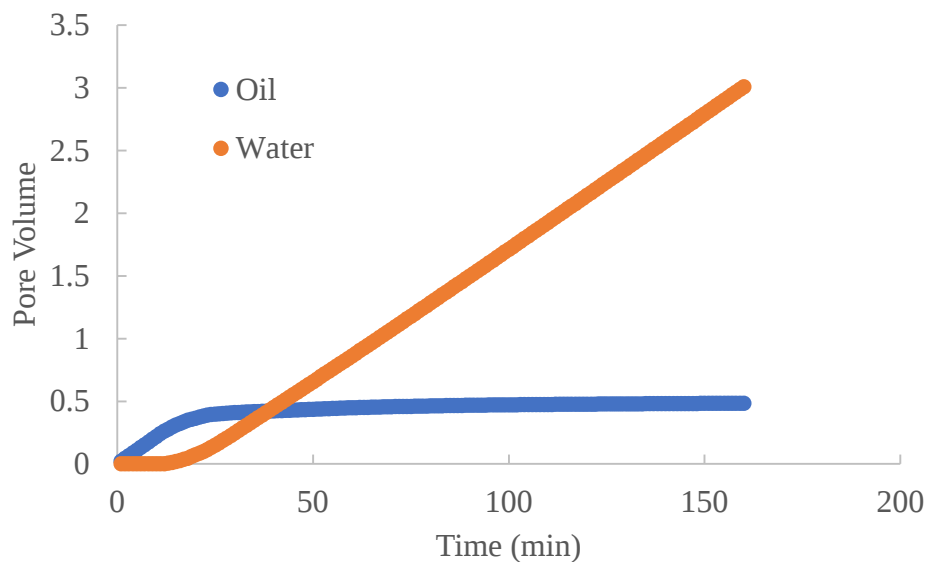


Figure 5.4 Flow-out pore volumes of water and oil during water flooding in water-wet sands.

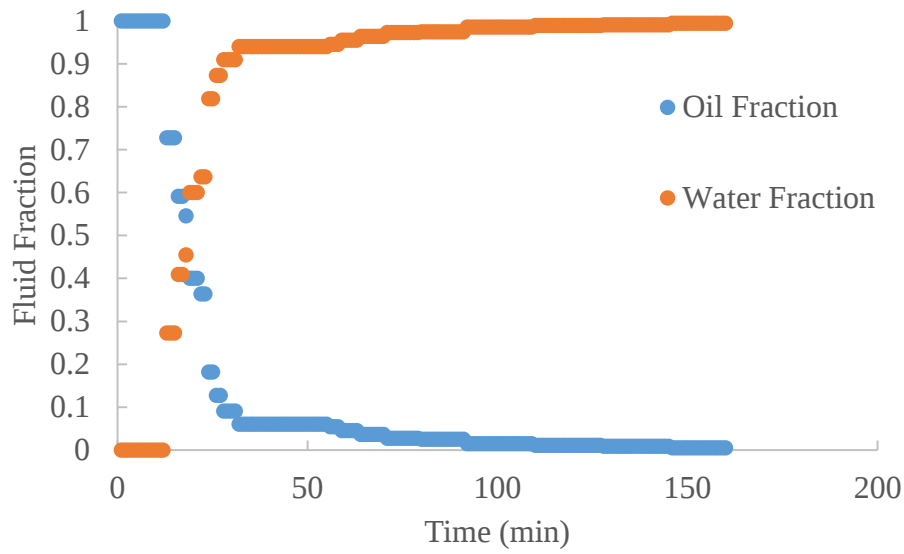


Figure 5.5 Flow-out fraction of water and oil during water flooding in water-wet sands.

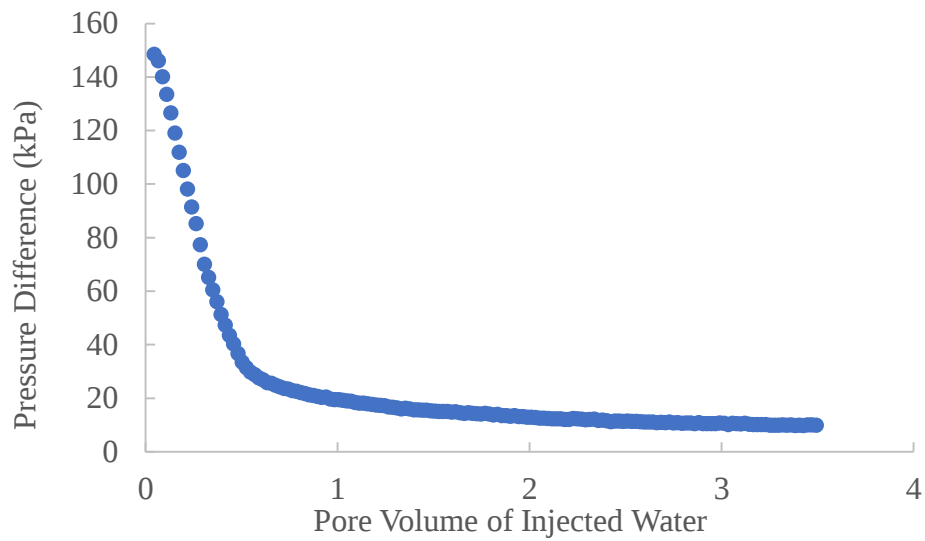


Figure 5.6 Pressure difference versus injected oil pore volume during water flooding in water-wet sands.

5.4 Water-wet Sand: Relative Permeability

5.4.1 Unsteady State Model Derivation

Here, the unsteady-state method is applied to calculate water-oil relative permeability curves as proposed by Johnson et al. (1959). The assumptions behind the unsteady state relative permeability method include: 1. the porous medium is homogeneous and incompressible, 2. the fluids are incompressible, and 3. the fluid properties are also constant during measurement. Because a high injection flow velocity is applied during measurement, a stabilized displacement front can be maintained and both the effects of capillary pressure and end effects can be neglected. Johnson et al.'s (1959) derivation of equations is based on the Buckley-Leverett Model after water breakthrough. The position, x , of a front with a given water saturation, S_w , is given by:

$$x = \frac{f'_w(S_w)}{\phi A} \int_0^t Q(t) dt \quad \text{Eq. 5-1}$$

where ϕ is the porosity of sand pack, $f'_w(S_w)$ is the derivative of the fractional flow with respect to water saturation, A is the cross-sectional area of measured sands, $Q(t)$ is the water injection rate, and t is the injection time. At the exit of the sand pack, Equation 5-1 becomes:

$$L = \frac{f'_w(S_{we})}{\phi A} \int_0^t Q(t) dt \quad \text{Eq. 5-2}$$

where L is the length of the sand pack, S_{we} is the water saturation at the exit of the sand pack and $f'_w(S_{we})$ is the derivative of the fractional flow with respect to the water saturation at the exit of the sand pack. The dimensionless pore volume of injected fluid Q_d :

$$Q_d = \frac{\int_0^t Q(t) dt}{\phi AL} = \frac{1}{f'_w(S_{we})} \quad \text{Eq. 5-3}$$

At a constant time, the pressure drop across the entire length of the sand pack is as follows:

$$\Delta P = - \int_0^L \frac{dP}{dx} dx \quad \text{Eq. 5-4}$$

where P is the fluid pressure and ΔP is the pressure difference. As known, the Darcy equation is given by:

$$\frac{Q_o}{A} = - \frac{kk_{ro}}{\mu_o} \frac{dP}{dx} \quad \text{Eq. 5-5}$$

where Q_o is the production oil rate, kk_{ro} is the oil phase effective permeability, and μ_o is the oil phase viscosity. The pressure gradient is then given by:

$$\frac{dP}{dx} = \frac{\mu_o Q_o}{Ak k_{ro}} = \frac{\mu_o Q f_o}{Ak k_{ro}} = \frac{\mu_o u f_o}{k k_{ro}} \quad \text{Eq. 5-6}$$

where Q is the total liquid production rate (both oil and water), f_o is the fractional flow with respect to oil, and u is the velocity of the water injection rate (assumes water and oil incompressibility within the sand pack). After combining Equations 5-4 and 5-6, the result is:

$$\Delta P = \frac{\mu_o u}{k} \int_0^L \frac{f_o}{k_{ro}} dx \quad \text{Eq. 5-7}$$

Combining Equations 1 and 2, the result is:

$$x = \frac{f'_w(S_w)}{f'_w(S_{we})} L \quad \text{Eq. 5-8}$$

and therefore,

$$dx = \frac{L}{f'_w(S_{we})} df'_w(S_w) \quad \text{Eq. 5-9}$$

by substituting Equation 5-9 into Equation 5-7, the result is:

$$\Delta P = \frac{\mu_o u}{k} \frac{L}{f'_w(S_{we})} \int_0^{f'_w(S_{we})} \frac{f_o}{k_{ro}} df'_w(S_w) \quad \text{Eq. 5-10}$$

or

$$\int_0^{f'_w(S_{we})} \frac{f_o}{k_{ro}} df'_w(S_w) = \frac{k \Delta P f'_w(S_{we})}{\mu_o u L} = \frac{f'_w(S_{we})}{I} = \frac{1}{Q_d I} \quad \text{Eq. 5-11}$$

and

$$I = \frac{\mu_o u L}{k \Delta P} = \frac{u / \Delta P}{k / (\mu_o L)} = \frac{u / \Delta P}{u_s / \Delta P_s} \quad \text{Eq. 5-12}$$

where u_s and ΔP_s are the water injection rate and pressure difference at the beginning of the water flooding experiment at the irreducible water saturation. We have:

$$\frac{d(\frac{1}{Q_d I})}{df'_w(S_{we})} = \frac{f_o(S_{we})}{k_{ro}(S_{we})} \quad \text{Eq. 5-13}$$

and

$$k_{ro}(S_{we}) = f_o(S_{we}) \frac{df'_w(S_{we})}{d\left(\frac{1}{Q_d l}\right)} = f_o(S_{we}) \frac{df'_w(S_{we})}{d\left(\frac{1}{Q_d l}\right)} = f_o(S_{we}) \frac{d\left(\frac{1}{Q_d}\right)}{d\left(\frac{1}{Q_d l}\right)} \quad \text{Eq. 5-14}$$

By using Darcy's equation at the exit of the sand pack, the water relative permeability is given by:

$$\frac{Q_w}{Q_o} = \frac{k_{rw}/\mu_w}{k_{ro}/\mu_o} = \frac{f_w Q}{f_o Q} = \frac{1-f_o(S_{we})}{f_o(S_{we})} \quad \text{Eq. 5-15}$$

and thus,

$$k_{rw}(S_{we}) = k_{ro}(S_{we}) \frac{\mu_w}{\mu_o} \frac{1-f_o(S_{we})}{f_o(S_{we})} \quad \text{Eq. 5-16}$$

Based on the Buckley-Leverett Model, the water saturation at the exit of the sand pack is:

$$S_{we} = \bar{S}_w - Q_d f_o(S_{we}) \quad \text{Eq. 5-17}$$

where $\bar{S}_w$ is the average water saturation in the sand pack. To sum up, the equations applied to calculate unsteady state relative permeability are as follows:

$$k_{ro}(S_{we}) = f_o(S_{we}) \frac{d\left(\frac{1}{Q_d}\right)}{d\left(\frac{1}{Q_d l}\right)} \quad \text{Eq. 5-14}$$

$$k_{rw}(S_{we}) = k_{ro}(S_{we}) \frac{\mu_w}{\mu_o} \frac{1-f_o(S_{we})}{f_o(S_{we})} \quad \text{Eq. 5-16}$$

$$S_{we} = \bar{S}_{we} - Q_d f_o(S_{we}) \quad \text{Eq. 5-17}$$

5.4.2 Water-wet Sands: Water-Oil Relative Permeability Curves

The k_{rw} at residual oil saturation can be easily calculated from Darcy's equation since at this situation the system reaches steady state with water only flow in the porous medium. The application of unsteady state method is relative straightforward, but calculating $d(1/Q_d)/d(1/Q_d I)$ in Equation 14 is more complex. Here, we first plot $(1/Q_d)$ versus $(1/Q_d I)$ as shown in Figure 5.7. Thereafter, a regression function is fitted to represent the relationship. The calculated relative permeability curves are shown in Figure 5.8. From the plot, the residual oil saturation is 0.41, and the corresponding k_{rw} is 0.23. This is the same as the k_{rw} calculated by Darcy equation when system reaches steady state in Chapter 5.3. This proves the accuracy of unsteady state method. Because the JBN method is used, the obtained relative permeability is normalized by the effective oil permeability of 7 Darcy at the irreducible water saturation.

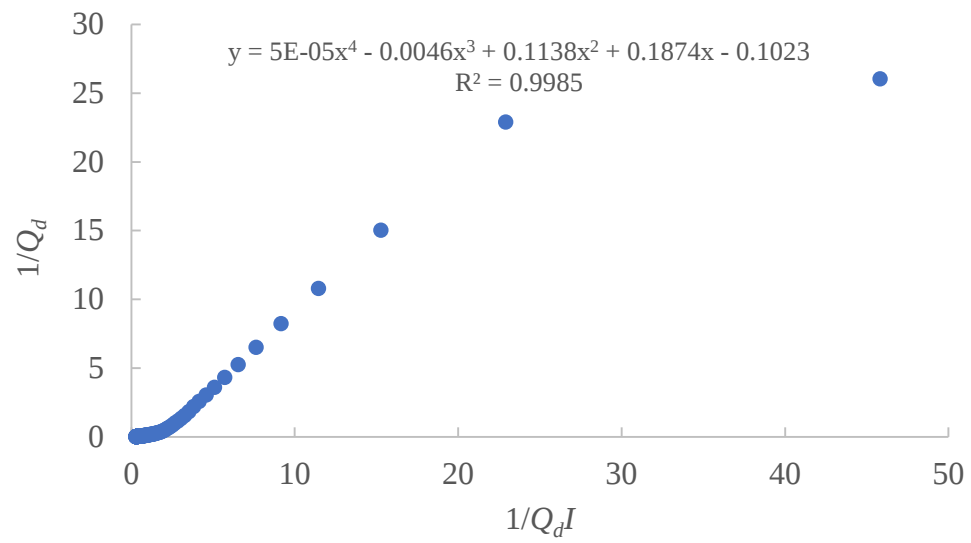
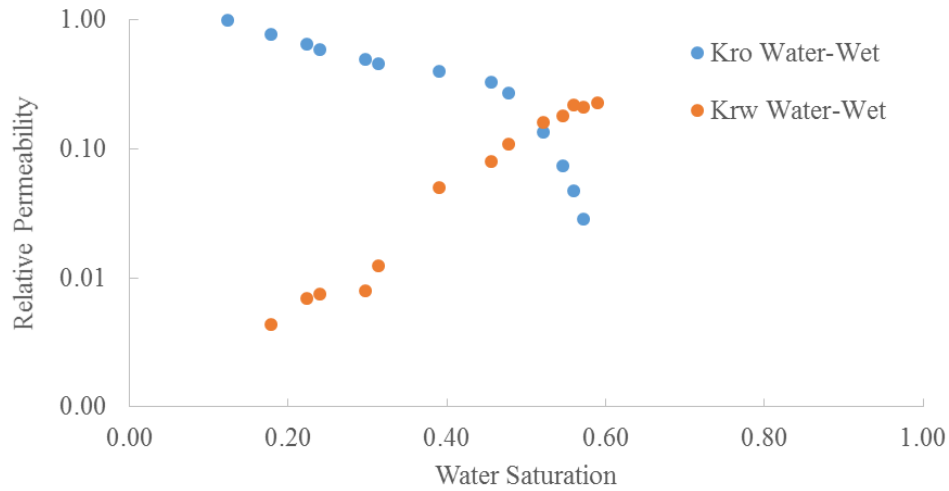


Figure 5.7 Water-wet sands: $1/Q_d$ versus $1/Q_d I$.

(a)



(b)

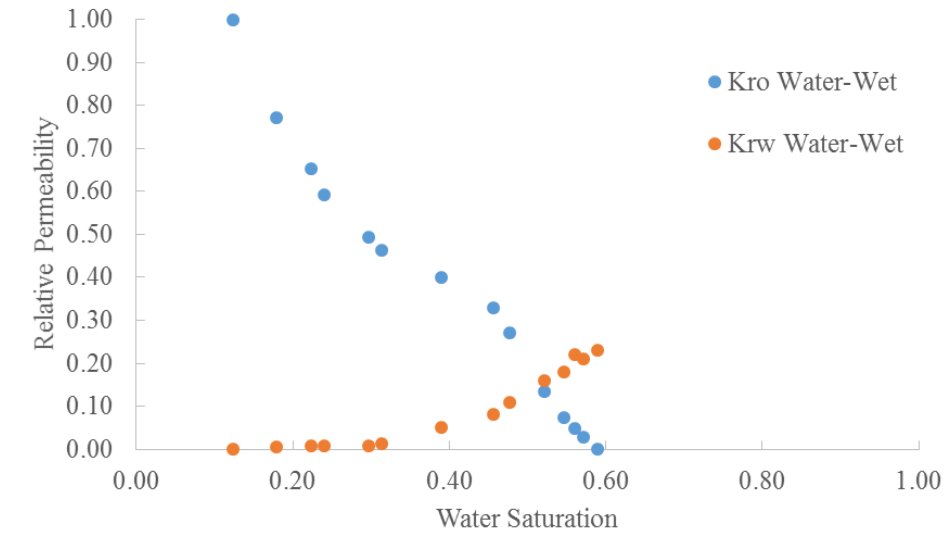


Figure 5.8 Water-oil relative permeability curves in water-wet sand calculated by the unsteady state method. (a) Relative permeability is displayed in normal scale; (b) relative permeability is displayed in log scale.

The relative permeability obtained by JBN is usually scattered. It is necessary that obtain smooth relative permeability for fluid flow calculation, no matter through a reservoir simulator or an

analytical model. We use following equations to fit smooth curves based on directly calculated points.

$$k_{rw}^* = k_{rw,max}(S_d)^{e_w} \quad \text{Eq.5-18}$$

$$k_{ro}^* = k_{ro,max}(1 - S_d)^{e_o} \quad \text{Eq.5-19}$$

$$S_d = \frac{S_w - S_{wi}}{1 - S_{wi} - S_{or}} \quad \text{Eq.5-20}$$

where k_{rw}^* is fitted water relative permeability, $k_{rw,max}$ is water relative permeability at residual oil saturation, k_{ro}^* is fitted oil relative permeability, $k_{ro,max}$ is oil relative permeability at irreducible water saturation, e_w exponent for water relative permeability, e_o exponent for oil relative permeability, S_d is normalized water saturation, S_w is water saturation, S_{wi} is irreducible water saturation, and S_{or} is residual oil saturation.

To fit Equations 5-18 and 5-19, the Microsoft excel solver is applied to minimize the sum error between fitted values and directly calculated values by tuning e_w for water relative permeability and e_o for oil relative permeability separately. The e_w and e_o for water wet sands are 2.7 and 1.1, respectively. Figure 5.9 displays the fitted oil-water relative permeability of water-wet sands.

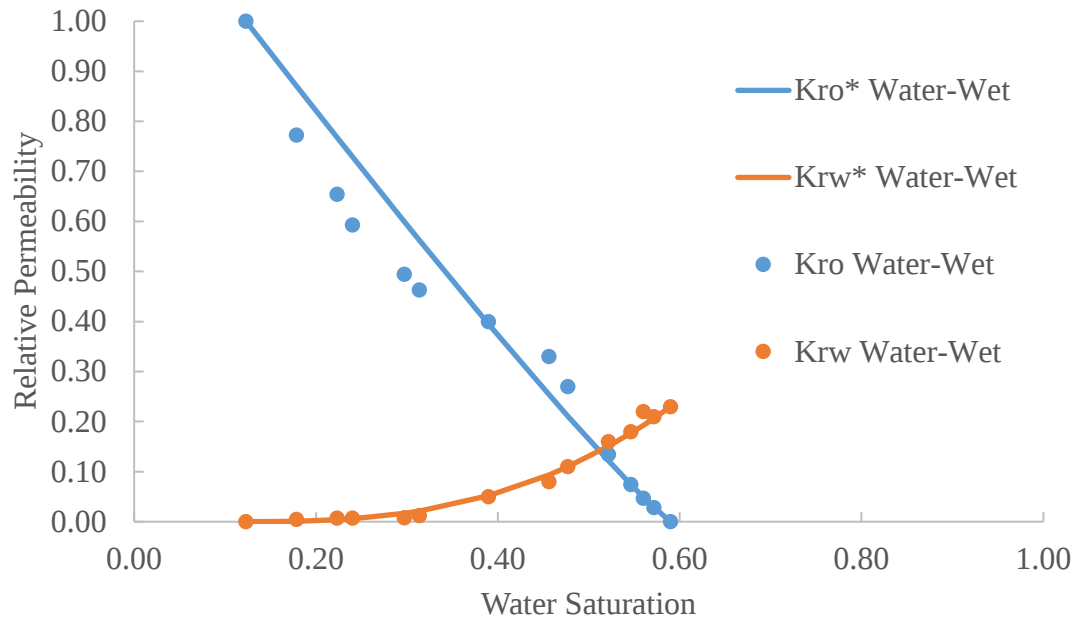


Figure 5.9 K_{ro}^* and K_{rw}^* are fitted oil and water relative permeability of water-wet sands, respectively. K_{ro} and K_{rw} are directly calculated oil and water relative permeability of water-wet sands, respectively.

The relative permeability can also reflect the sands wettability. The water saturation at which oil and water relative permeabilities are equal is over 50%, and k_{rw} at the maximum water saturation (residual oil saturation) is less than 30%. Based on Craig (1971)'s work, these results suggest that the sands are water-wet.

5.5 Oil-wet Sand: Absolute Permeability Measurements by Oil Injection

Since the oil-wet sands cannot be fully saturated with water, oil is used in single-phase flow. Oil is injected at constant rate into the sand and the pressure difference and production rate of oil at

the exit of the sand pack is measured. As described above in Section 5.1, the absolute permeability is determined from a set of data obtained at multiple flow rates and pressure differences. Here, twenty absolute measurement experiments are conducted. The oil injection flow rates range from 3.8 ml/min to 5.7 ml/min. During each measurement, the pressure difference was measured after the system reached steady state. From the data, the average value of the absolute permeability was found equal to 7.5 D. Linear regression of all of the data plotted in Figure 5.10, yields an absolute permeability of 7.4 D with a correlation coefficient equal to 0.97.

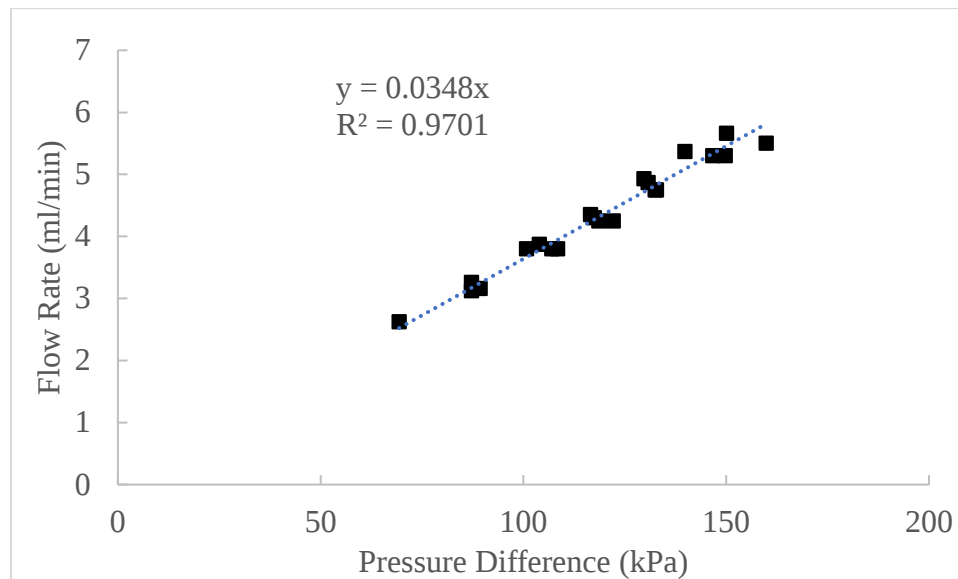


Figure 5.10 Relationships between flow rates and pressure differences during absolute permeability measurements of oil-wet sands.

5.6 Oil-wet Sand: Restoring Reservoir Conditions by Water and then Mineral Oil Injection

There are two steps to restore the reservoir conditions. In the first step, water is injected into oil-wet sands that has been fully saturated with the mineral oil. The water injection rate is set constant

at 6 ml/min and is continued until the production water cut is greater than 99.5%. Figure 5.11 shows the production volumes of water and oil. The data reveals that water breakthrough occurs at 7 minutes. Figure 5.12 displays that the pressure difference severely drops before water breakthrough and then declines at a slower rate as the experiment proceeds.

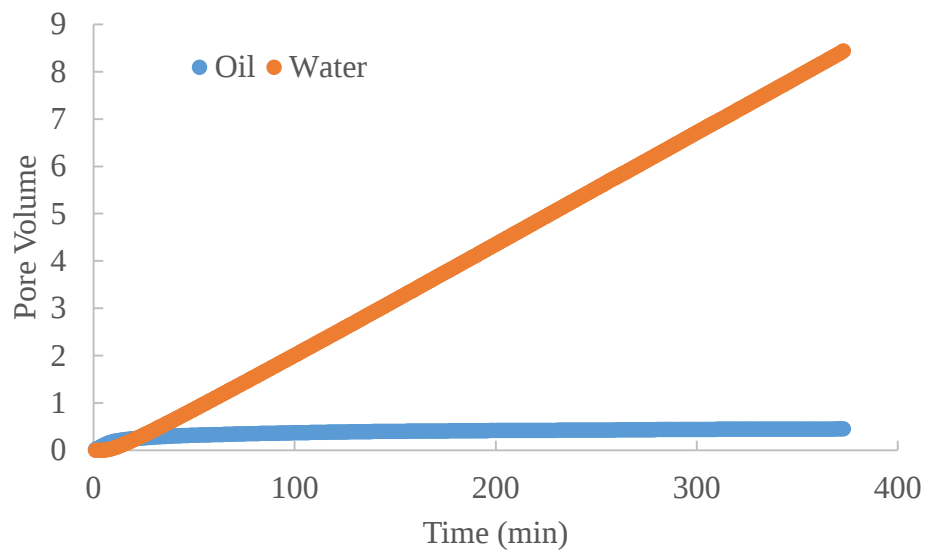


Figure 5.11 Flow-out pore volumes of water and oil during water injection in oil-wet sands.

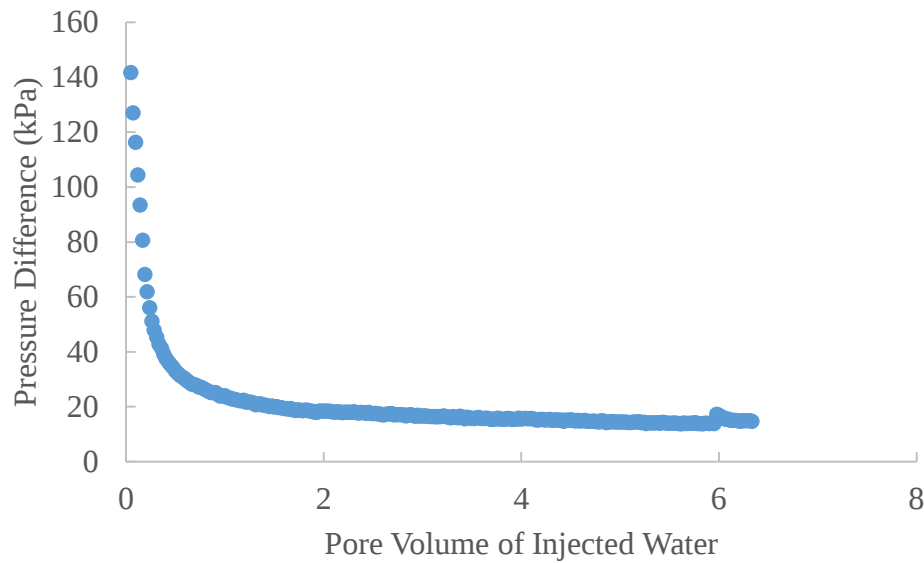


Figure 5.12 Pressure difference versus injected water pore volume during water injection in oil-wet sands.

In the second step, mineral oil is injected at 5.5 ml/min into the oil-wet sand pack until the production oil cut is 100%. The results are displayed in Figure 5.13. The breakthrough time is observed to occur at 20 minutes. After breakthrough, the water volume increases slightly and the production water cut is less than 1%. After 41 minutes of oil injection, no produced water can be observed.

Figure 5.14 displays the relationship between the pore volumes of injected oil and pressure drop. The pressure drop builds up as injection occurs since the increasing saturation of high viscosity oil results in a higher pressure requirement to maintain a constant oil injection rate. A bigger jump occurs at the time of oil breakthrough, since an oil flow path has now been established in the sand pack and relatively little water can flow from the pack. Thereafter, the pressure difference increases at a very slow rate due to the very small additional amounts of oil being accumulated in porous

medium after breakthrough. From a calculation of the mass balance on the water and oil, the irreducible water saturation equal to 4% is determined. An oil effective permeability of 5.6 Darcy at irreducible water saturation is calculated by using the Darcy equation after the system a quasi-steady state condition and only oil flows into and out from the system.

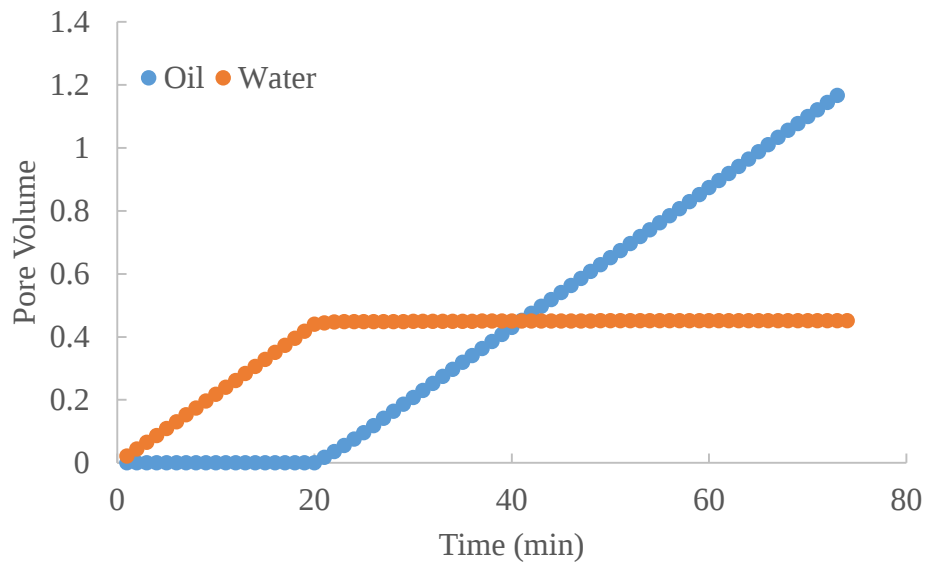


Figure 5.13 Flow-out pore volumes of water and oil during oil injection in oil-wet sands.

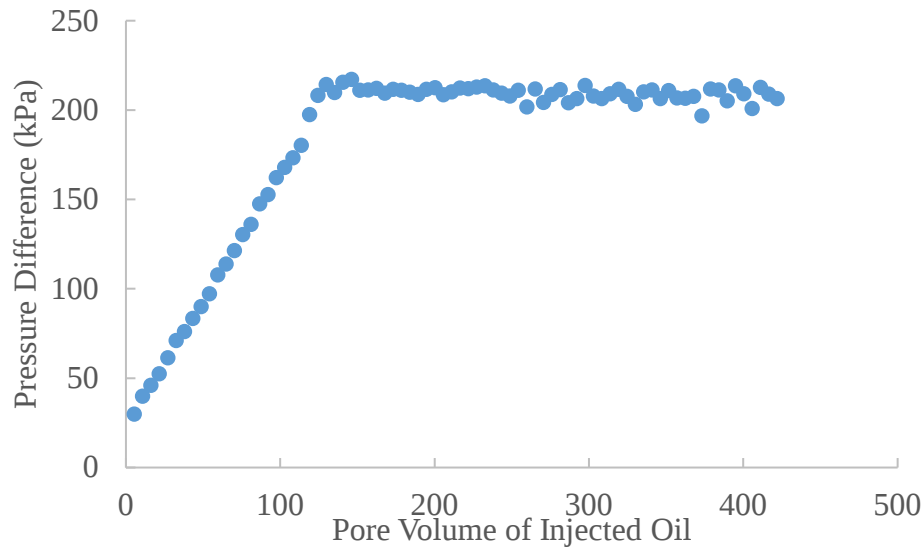


Figure 5.14 Pressure difference versus injected oil pore volume during oil injection in oil-wet sands.

5.7 Oil-wet Sand: Water Flooding

After saturating the pack with mineral oil, during water-flooding of the oil-wet sand, a constant water flow-in rate of 6 ml/min is applied. The injection stops when the production water cut is over 99.5%. Figure 5.15 and Figure 5.16 display that water breakthrough occurs after 6 minutes of injection. Figure 5.17 shows that the pressure difference severely decreases before water breakthrough and then decreases further at a more gradual rate. The oil recovery factor at water breakthrough is equal to 13% and that the oil ultimate recovery factor is equal to 52%. The water relative permeability of 0.61 at residual oil saturation of 0.51 is calculated by using the Darcy equation after the system a quasi-steady state condition and only water flows into and out from the system.

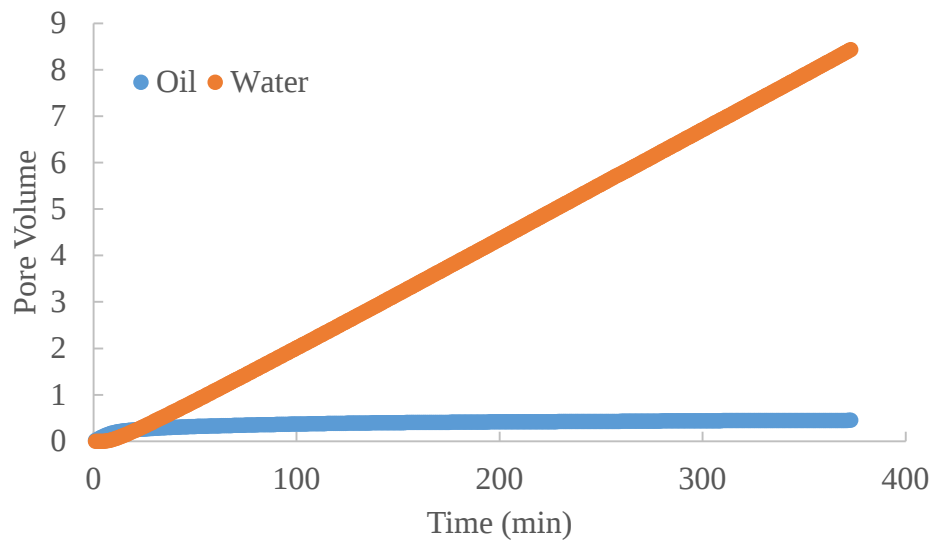


Figure 5.15 Flow-out pore volumes of water and oil during water flooding in oil-wet sands.

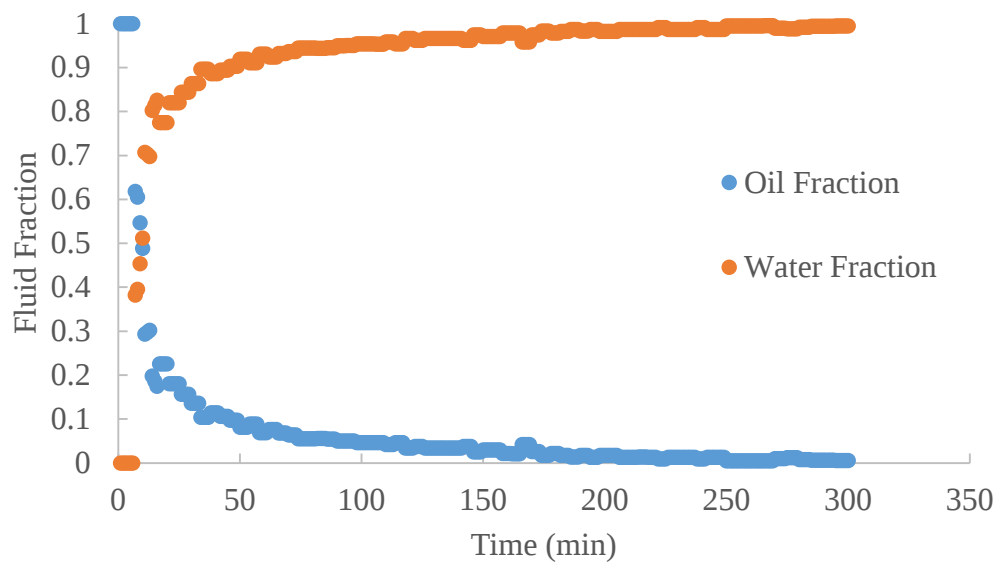


Figure 5.16 Flow-out fraction of water and oil during water flooding in oil-wet sands.

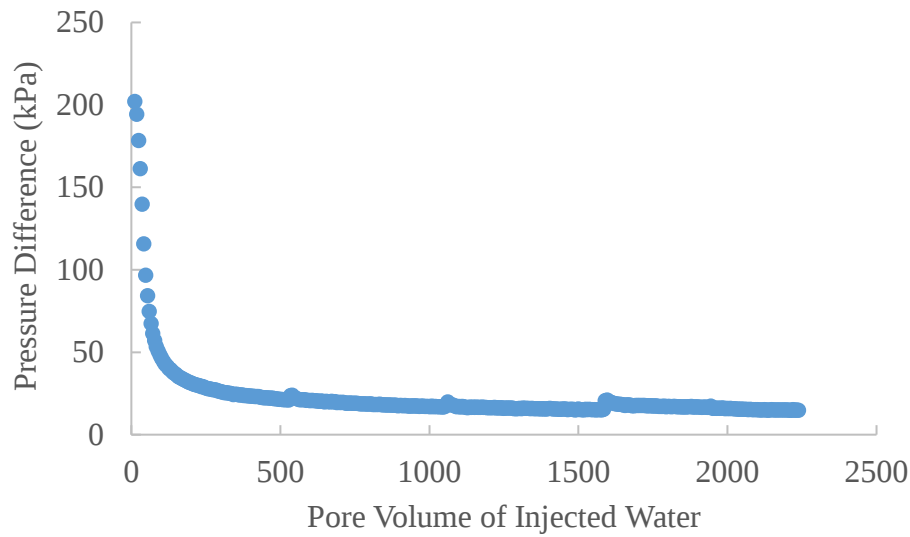


Figure 5.17 Pressure difference versus injected water pore volume during water injection in oil-wet sands.

5.8 Oil-wet Sand: Relative Permeability

The water-oil relative permeability of oil-wet sands is calculated the unsteady state method as stated in the Section 5.4.1. The plot of $1/Q_d$ versus $1/Q_d I$ is displayed in Figure 5.18. The curves were regressed to obtain values of $d(1/Q_d)/d(1/Q_d I)$. Figure 5.19 shows the water-oil relative permeability curves obtained for the oil-wet sands. The residual oil saturation is equal to 0.51, and the corresponding k_{rw} is 0.61. The water saturation at which oil and water relative permeabilities are equal is 31% (less than 50%), and k_{rw} at the maximum water saturation (residual oil saturation) is greater than 30%. The connate water saturation is less than 10%. Based on Craig (1971)'s work, these phenomena prove that the tested sands are oil-wet. The obtained relative permeability is normalized by the effective oil permeability of 5.6 Darcy at the irreducible water saturation.

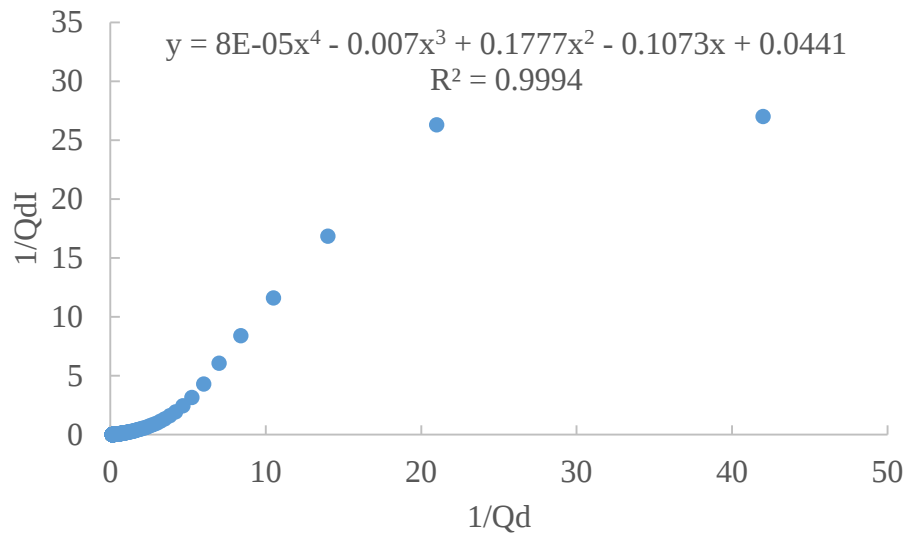


Figure 5.18 Oil-wet sands: $1/Q_d$ versus $1/Q_d I$.

To obtain smooth relative permeability curves, the same fitting process stated in Section 5.4.2 is applied. The fitted e_w and e_o for oil-wet sands are 3.1 and 1.9, respectively. Figure 5.20 displays the fitted oil-water relative permeability of water-wet sands.

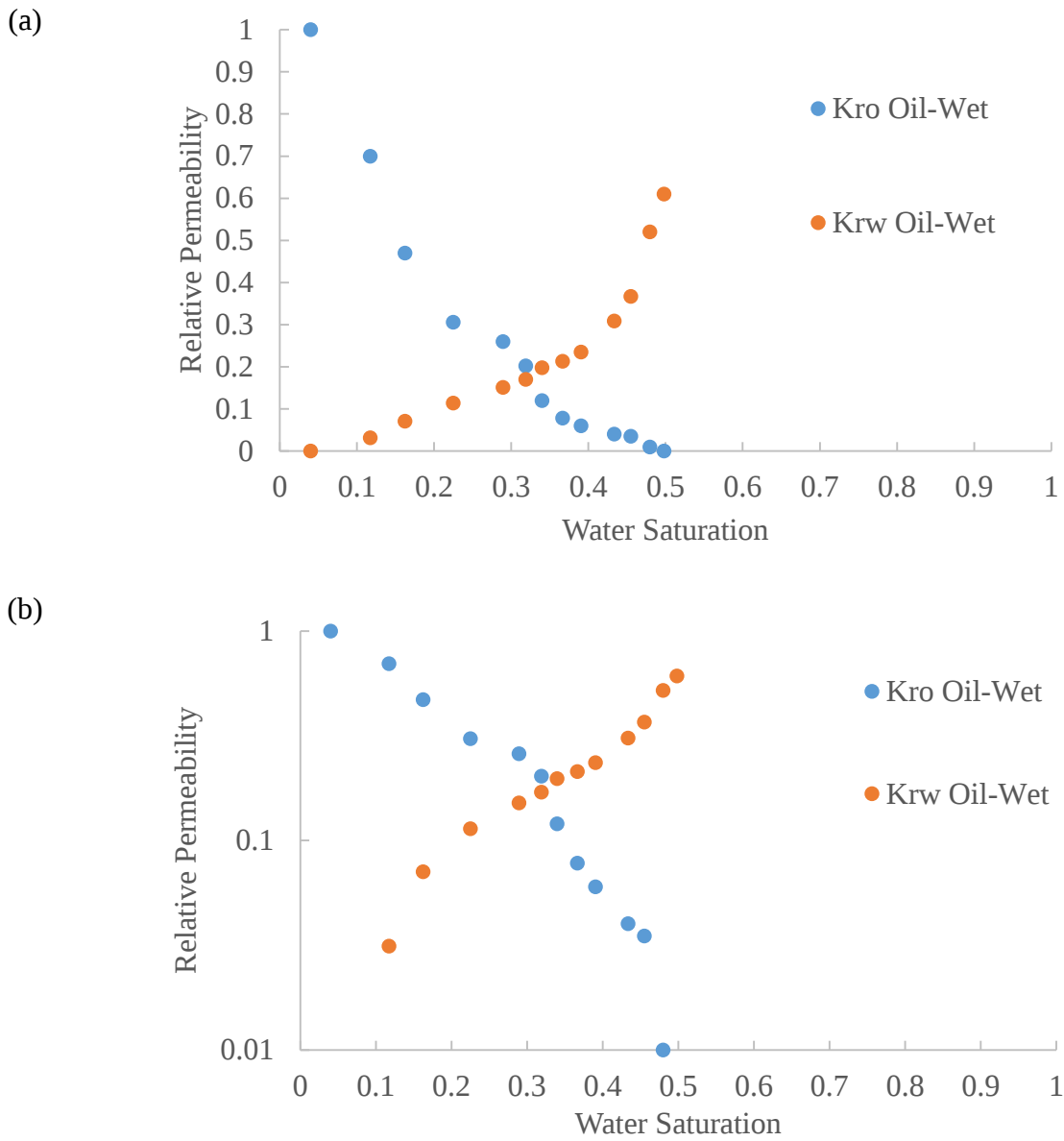


Figure 5.19 Water-oil relative permeability of oil-wet sands. (a) Relative permeability is displayed in normal scale; (b) relative permeability is displayed in log scale.

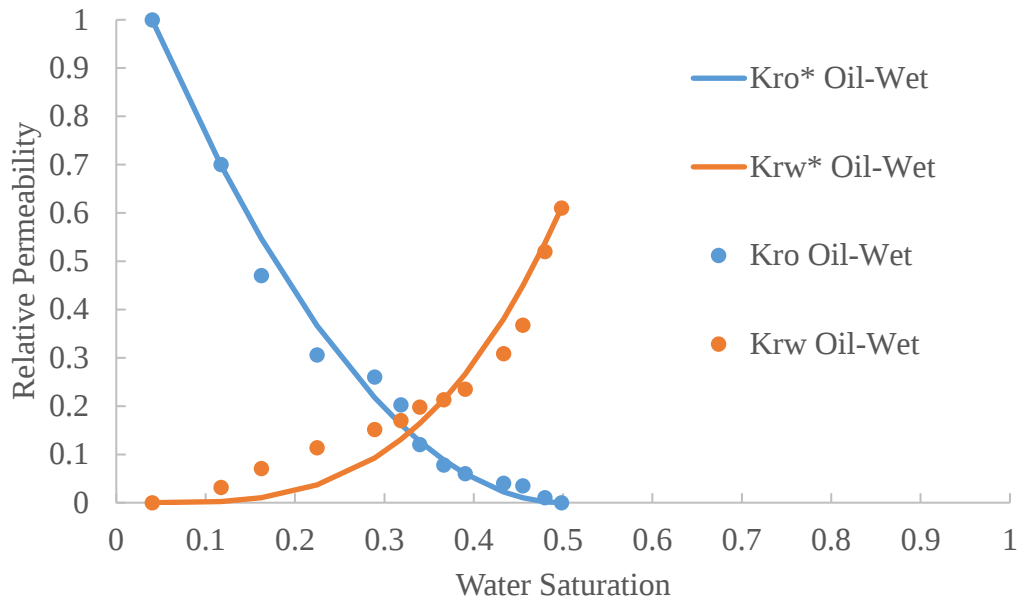


Figure 5.20 K_{ro}^* and K_{rw}^* are fitted oil and water relative permeability of water-wet sands, respectively. K_{ro} and K_{rw} are directly calculated oil and water relative permeability of water-wet sands, respectively.

5.9 Discussion of Results

As stated in Chapter 4, McMurray Formation oil sands have been used to prepare both water-wet sands and oil-wet sands. In this Chapter, multiphase flow experiments were conducted in both water-wet and oil-wet sands by water displacement to obtain relative permeability curves.

The most obvious differences between the water-wet and oil-wet relative permeability curves are the irreducible water saturation and residual oil saturation and their corresponding water and oil permeabilities. The oil effective permeability of 7 Darcy, at irreducible water saturation of 0.12,

for the water-wet sands is larger than the value of 5.6 Darcy, at irreducible water saturation of 0.04, for the oil-wet sands. It indicates that at the irreducible water saturation, the oil phase is distributed in the larger pores of water-wet sands and smaller pores of the oil-wet sands, which explains the higher oil phase conductivity in water-wet sands. The water effective permeability of 1.6 Darcy, at residual oil saturation of 0.41, for the water-wet sand is smaller than the value of 3.4 Darcy, at residual oil saturation of 0.51, for the oil-wet sand. This indicates the water phase is distributed in the smaller pores of the water-wet sands and larger pores of oil-wet sands, which explains the higher water phase conductivity in the oil-wet sands.

In terms of the relative permeability curves, the differences are also very obvious, as shown in Figure 5.21. At a given saturation, the oil relative permeability in the water-wet sands is larger than that of the oil-wet sands, whereas the water relative permeability of the water-wet sands is smaller than that of the oil-wet sands. These comparisons show that oil phase flows well in the water-wet sands, but after wettability alternation from water-wet to oil-wet, the oil phase is more likely trapped in the porous media.

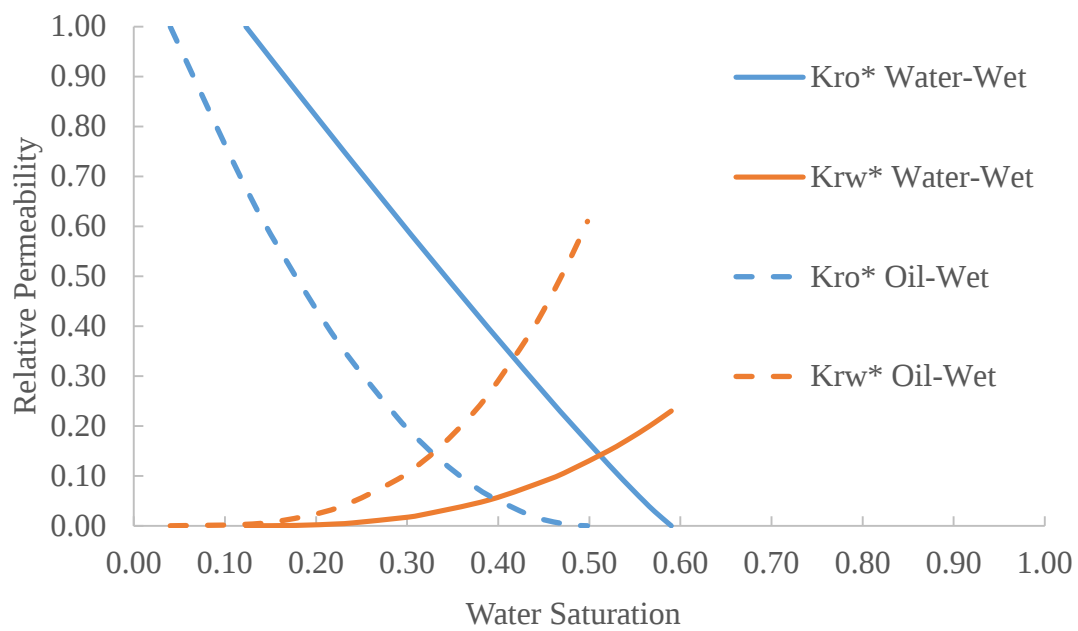


Figure 5.21 Relative permeability curves comparison between water-wet and oil-wet sands.

Chapter Six: Conclusions and Recommendations

6.1 Conclusions

The unconsolidated McMurray Formation oil sands in clean state are considered to be water-wet and the experiments conducted here prove this. However, bitumen is a strong wettability alteration agent given its large content of resin and asphaltenes. After the sands grains are directly exposed to resin and asphaltenes, their polar ends attach on the grains and the hydrocarbon ends are exposed to fluid. Therefore, the wettability of sand grains is altered from water-wet to oil-wet. The dissolution of asphaltenes in toluene helps to evenly adsorb asphaltenes on the surface of the sand grains and the high temperature drying process enhances the coating of asphaltenes on grains. As a result, strong oil-wet sands have been produced in the method devised in this research. This process for generating oil-wet sand is novel and is a major contribution of the research reported in this thesis.

Water displacement experiments on water-wet and oil-wet sands were conducted. The unsteady state method was applied to calculate the oil and water relative permeability curves in these systems. The results show that the two relative permeability curves show significant differences. The k_{rw} at residual oil saturation of water-wet sands and oil-wet sands is 0.21 and 0.61, respectively. The water saturation at cross-point ($k_{rw} = k_{ro}$) of water-wet sands is 54%, while the one of oil-wet sands is 31%. These features are similar with the typical water-wet and oil-wet relative permeability stated by Craig (1971). Although the connate water saturation (12%) of water-wet sands is higher than the one (4%) of oil-wet sands, connate water saturation of water-wet

sands is still lower than the ones of typical sandstones (water saturation over 20%). The reason is that the measured samples are unconsolidated sandstones, and the diameter of pore media is large, a smaller irreducible water content can be preserved in examples. The results further confirm the change of the wettability of the sands obtained from the high temperature adsorption process. Contact angle measurements also support the results from the relative permeability experiments.

The research documented in this thesis provides an insight into how wettability can be changed and the effects of wettability on multiphase flow in the McMurray Formation oil sand. When the grains are not contaminated by bitumen, they are water-wet. Water-wet relative permeability should be used for the design of reservoir simulation or design. If grains have been contaminated with bitumen, they can be oil-wet especially if the temperature is elevated. This can be the case in steam-based and air injection based recovery processes. In these cases, oil-wet relative permeability curves should be applied. The change of wettability of oil sands reservoir rock at elevated temperature in the presence of asphaltenes has not been reported before.

6.2 Recommendations

We have prepared oil-wet and water-wet sands by using oil sands and bitumen, but a more sophisticated measurement is still required to investigate how much asphaltenes is adsorbed on grains. In this way, we can qualify the requirement amount of asphaltenes to alter wettability. Although effects of wettability on multiphase flow have been investigated, we only conducted water displacement under room temperature. In the future study, a high temperature conduction should be conducted to investigate effects of temperature on wettability and relative permeability.

The future study should also investigate effects of capillary pressure on displacement tests. It is recommended that further studies are conducted to understand on how asphaltenes adsorb on quartz sand grains and how this depends on temperature. This should also be done in the presence of steam.

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