Conformance Control for Enhanced Oil Recovery in Fractured Reservoirs

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Summary

Fractures in a hydrocarbon reservoir often exhibit permeabilities several orders of magnitude higher than the rock matrix, and heavily influence fluid flow. Injected fluids and gases may channel through the fractures without contacting large volumes of matrix oil, which cause poor microscopic and macroscopic sweep efficiencies, and hence low oil recovery factors.

Secondary recovery methods, which include the injection of water or gas into the reservoir, may be successful in fractured reservoirs if 1) the wettability is such that it enables capillary spontaneous imbibition of water (during waterfloods), or 2) if matrix blocks are in capillary contact with each other, so that fluids and viscous pressure gradients can be transported across fractures (water and gas floods). During gas floods, gravity drainage is an important recovery mechanism, and efficient recovery of oil may be obtained at gravity stable conditions in matrix blocks of large continuous column heights. In severely fractured reservoirs, without continuity between matrix blocks, high adverse mobility ratios and large density contrast between injected gas and reservoir oil may lead to fluid channeling and poor macroscopic sweep efficiency. Fluid channeling due to fractures may also be severe during waterfloods in reservoirs with low viscous and capillary contributions to flow. Enhanced oil recovery (EOR) methods may be applied to improve the displacement of matrix oil, by e.g. reducing interfacial tension and capillary contrasts, and/or by increasing the viscosity of the displacing fluid. The main challenge during EOR operations in fractured reservoirs is to prevent fracture channeling, so that injected fluids and gases can contact and displace the oil in the matrix. Conformance control methods reduce the impact of fractures on fluid flow and enable fluids to enter and flood the matrix. Two methods were experimentally investigated in this thesis: 1) polymer gel injection to reduce fracture permeability or transmissibility, thus fluids are enabled to enter the matrix at increased viscous pressure gradients during chase floods (**Paper 1-2** and **Paper 5**), and 2) foam injection to reduce gas mobility. Foam injection improve microscopic and macroscopic sweep during gas floods, by increasing the gas viscosity and the resistance to flow in fractures (**Paper 3-5**).

Conformance control by polymer gel injection was experimentally assessed through laboratory core floods. In **Paper 1**, two gel injection methods and their implications on gel strength during chase floods were investigated: 1) immature gel (gelant) injection, with following *in-situ* gelation, and 2) injection of fully formed, mature gel. Gel propagation through fractured systems is fundamentally different for the two. Immature gel flows in porous rock as well as fractures, hence a resilient gel form in the fracture and surrounding matrix after injection and gelation. Mature gel is inhibited from entering porous rock due to its structure and flows through fractures only: the gel may, however, lose water and

consequently become more concentrated during injection, which improves its strength. The rupture pressure, where the gel in a fracture breaks and allows fluid transport through it, was measured and found to be comparable for both gel placement methods. After breaking, gel was observed to significantly reduce fracture permeability during chase waterfloods, with factors of 5000 (mature gel) and 600 (immature gel) compared to the initial fracture permeability. Permeability reduction provided by mature gel was stable during injection of over 100 fracture volumes (FV) of water through the fracture.

Polymer gel behaviour is often studied using cores saturated by water only, including the work in **Paper 1**. In **Paper 2-6**, experiments were performed using core plugs saturated by two phases: water and oil. Saturation by more than one phase yield saturation functions that influence fluid flow in the matrix, and in the case of immiscible fluids, capillary forces may impact the fluid dynamics. In **Paper 2**, capillary forces in strongly water-wet matrices attracted water from mature polymer gel by spontaneous imbibition. Spontaneous imbibition was observed in three different outcrop core materials, and the oil recovery rate as a function of time was measured when all core surfaces were exposed to gel; using all faces open (AFO) boundary conditions, and when the core end faces only were open to flow, applying two ends open- free spontaneous imbibition (TEOFSI) boundary conditions, where one end face was in contact with gel and one with oil. Severe shrinkage of the gel volume was observed due to spontaneous imbibition of gel solvent, and was measured to be up to 99%. Shrinkage at this level can be detrimental for the gels ability to efficiently block fractures. Capillary spontaneous imbibition and subsequent gel shrinkage may help explain why gel treatments have lost efficiency with time when applied in water-wet reservoirs.

Paper 3 investigated miscible CO_2 EOR in fractured and un-fractured systems. Laboratory core floods were performed at reservoir conditions, and CO_2 was injected in the secondary or tertiary recovery mode. Oil composition significantly influenced the gas flood efficiency during the experiments, and secondary CO_2 floods recovered 96%OOIP when injected into core plugs saturated with simple mineral oil (n-Decane), within 2 to 4 pore volumes (PV) CO_2 injected (whole and fractured core plugs, respectively). Using North Sea crude oil as the oil phase, tail production was significant and several pore volumes were required to reach the residual oil saturation in both whole and fractured core plugs. The need for conformance control and increased viscous pressure gradients during gas floods was obvious, and CO_2-foam applications were investigated through core floods in **Paper 4**. Foam was applied to fractured core plugs in tertiary mode, at both miscible and immiscible experimental conditions. One objective was to enhance oil recovery at oil-wet conditions. Waterfloods were inefficient in recovering oil from fractured, oil-wet core plugs (0-22%OOIP) and the potential for CO_2 EOR was high. Miscible foam injection was the most efficient application, and recovered an additional 50-70%OOIP within 2PV foam injected, compared to 35%OOIP in 5PV for pure, miscible CO_2. Immiscible CO_2 foam injection was not efficient, and required over 30PV injected to reach the end point (15-20%OOIP). *In-situ* imaging by CT was used to investigate EOR by immiscible foam in fractured core plugs, and we found that gas did not enter the matrix at oil-wet conditions, but only flowed in fractures. Oil was displaced by surfactant solution, which entered the matrix at an increased pressure gradient provided by the foam.

Paper 5 combined polymer gel and CO_2-foam floods for integrated enhanced oil recovery (IEOR) in fractured, oil-wet core plugs. The first step in the integrated approach was conformance control by polymer gel. Chase flooding followed, and CO_2-foam floods were compared to chase floods by water and surfactant. CO_2-foam was observed to give a more stable displacement front during chase flooding and was less prone to viscous fingering. All experimental steps were monitored by *in-situ* imaging.

Two-phase flow functions were important during gel placements and chase floods in two-phase saturated media. Accurate methods to measure and analyze capillary pressure and relative permeability are always in demand. **Paper 6** presents an *in-situ* approach for measuring saturation and capillary pressure during waterfloods. Relative permeability was calculated from the experimental data using an explicit method.

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List of scientific papers

1. **Brattekås, B.**, Pedersen, S.G., Nistov, H.T., Haugen, Å., Graue, A., Liang, J. and Seright, R.S.: *"The Effect of Cr(III) Acetate-HPAM Gel Maturity on Washout from Open Fractures"*, SPE 169064, proceedings at the 2014 SPE Improved Oil Recovery Symposium held in Tulsa, Oklahoma, USA, 12 – 16 April 2014.

2. **Brattekås, B.**, Haugen, Å., Graue, A. and Seright, R.S.: *"Gel Dehydration by Spontaneous Imbibition of Brine from Aged Polymer Gel"*, SPE 153118, SPE Journal, vol. 19, issue 01, pp. 122 – 134, February 2014

3. Steinsbø, M., **Brattekås, B.**, Ersland, G., Fernø, M. and Graue, A.: *«Supercritical CO_2 injection for enhanced oil recovery in fractured chalk»*. Presented at the International Symposium of the Society of Core Analysts held in Avignon, France, 7-12 September 2014.

4. Haugen, Å., Mani, N., Svenningsen, S., **Brattekås, B.**, Graue, A., Ersland, G. and Fernø, M.: *"Miscible and Immiscible Foam Injection for Mobility Control and EOR in Fractured Oil-Wet Carbonate Rocks"*, Transport in Porous Media (TiPM), published online May 14th, 2014.

5. **Brattekås, B.**, Haugen, Å., Ersland, G., Eide, Ø., Graue, A. and Fernø, M.A.: "*Fracture Mobility Control by Polymer Gel- Integrated EOR in fractured, Oil-Wet Carbonate Rocks"*, SPE 164906, proceedings at the EAGE Annual Conference & Exhibition incorporating SPE Europec held in London, United Kingdom, 10–13 June 2013.

6. **Brattekås, B.**, Brautaset, A., Haugen, Å. and Graue, A.*: "Direct calculation of dynamic relative permeabilities from in-situ phase pressures and fluid saturations"*, SCA 2013-097, Peer-reviewed conference proceedings, International Symposium of the Society of Core Analysts held in Napa Valley, California, USA, 16 – 19 September 2013.

1. An introduction to fractured reservoirs

More than 20% of the world hydrocarbon reserves are contained in fractured reservoirs, often consisting of carbonate rock material (Saidi 1987, Firoozabadi 2000). Fractures, when present in a reservoir, significantly influence fluid flow and lead to a lower and slower recovery of hydrocarbons compared to non-fractured reservoirs. Naturally occurring fractures are often caused by brittle failure due to geological processes (folding, faulting, release of overburden pressure), and can exist in any type of rock (Miller 2010). Drilling and fluid injection in hydrocarbon reservoirs induce variations in temperature and/or pressure that may also cause intentional or unintentional fracturing of the reservoir rock.

It can be useful to categorize fractured reservoirs, to predict recovery and conformance challenges and to correctly model the reservoirs. A typology based on the relationship between porosity and permeability is given in **Table 1**, based on the characterizations of Nelson (2001) and Allen and Sun (2003). The recovery methods assessed in this thesis concentrate on Type 2 and Type 3 reservoirs, where the main hydrocarbon storage is in the matrix.

Table 1: Naturally fractured reservoirs typology.

	Matrix		Influence of fractures
	Porosity (storage)	**Permeability (productivity)**	
Type 1	little to none	little to none	The interconnected fracture network constitutes the hydrocarbon storage and controls the fluid flow.
Type 2	low	low	Fractures control the fluid flow. Fracture intensity and distribution dictates production. Some hydrocarbons are stored in the matrix.
Type 3	high	low	Matrix provides storage capacity, while the fractures augment permeability and transports hydrocarbons to producing wells.
Type 4	high	high	The effects of the fracture network are less significant on fluid flow. Fractures contribute to neither permeability nor porosity, but add significant reservoir heterogeneity.

There are fundamental differences between fluid flow performance in fractured and conventional, non-fractured, reservoirs. Van Golf-Racht (1996) described several, some of which are summarized below:

- *The pressure drop around a producing well in a fractured reservoir is low*. Even for high well injection rates, significant pressure drops are not experienced due to the high permeability of the fractures. Thus, the resulting low pressure gradient is sufficient for transportation of oil through fractures, but is too low to promote an exchange of fluids between the matrix and fracture network. The production of oil from matrix blocks is controlled by other production mechanisms that are specific for fractured reservoirs, e.g. spontaneous imbibition and gravity drainage.

- *Fluid transition zones are absent.* Distinct, horizontal interfaces separate the water-oil and oil-gas contacts at both static and dynamic conditions, rather than large transition zones as seen in non-fractured reservoirs.
- *The producing gas-oil ratio is substantially lower in fractured reservoirs*. Good vertical communication due to high permeable fractures cause liberated gas to segregate towards the top of the reservoir.
- *The rate of pressure decline per unit oil produced is normally low.* Production mechanisms in fractured reservoirs assure a great supply of fluids from matrix toward fractures (gravity and imbibition combined with fluid expansion, segregation, convection etc.). Comparable decline can be achieved in conventional reservoirs by re-injecting up to 80% of the produced gas.

1.1. Fluid flow in fractured reservoirs

Fluid flow is governed by gravitational, viscous and capillary forces. In fractured reservoirs, viscous forces are generally limited, and oil recovery is dominated by gravity and/or capillary forces, depending on matrix wettability (Boerrigter *et al.* 2007). The difference in capillary pressure between the matrix and fracture, in particular, has a significant effect on oil recovery (Firoozabadi 2000). Important production mechanisms that control oil recovery from matrix blocks in fractured reservoirs rely largely on capillary pressure, and include spontaneous imbibition and gravity drainage. Some factors that influence recovery mechanisms and performance are described below.

Key factors

Wettability

Wettability controls the location, distribution and flow of fluids in a porous matrix and influence the shape of the relative permeability and capillary pressure curves (Anderson 1987a, 1987b, 1987d). The wettability of a solid surface, contacted by two or more immiscible fluids, is defined by how the fluids spread on, or adhere to, the surface (Craig 1971). The wettability of a simple mineral surface is illustrated in **Figure 1**. Fluid flow in fractured reservoirs is heavily influenced by wettability. At water-wet conditions, a positive capillary pressure exists and water spread on pore surfaces in the presence of oil. The positive capillary pressure drives water invasion into matrix blocks by spontaneous imbibition. At oil-wet conditions, the capillary pressure is negative and oil is the spreading fluid. Water can therefore not enter an oil-wet matrix block without a viscous pressure gradient (Fernø *et al.* 2008, Haugen *et al.* 2010b).

Rock that has not been contacted by oil is expected to be strongly water-wet, e.g. outcrop rock, small water saturated pores and zones in a reservoir which have not been invaded by oil. Deposition of wettability-altering components from crude oil on a mineral surface can change its wetting preference (Buckley *et al.* 1998). The pattern of deposition and wettability alteration in the pore space depends on the distribution of the initial water saturation (Salathiel 1973). Most reservoirs are accepted to have some form of mixed wettability, where the wettability distribution often depends on pore size. Salathiel's (1973) mixed wettability refers to systems where oil-wet surfaces are continuous through the larger pores, where crude oil is envisioned to migrate through a reservoir. The small pores, where the capillary threshold pressure is too high for crude oil to enter during migration, remain water-wet and water saturated.

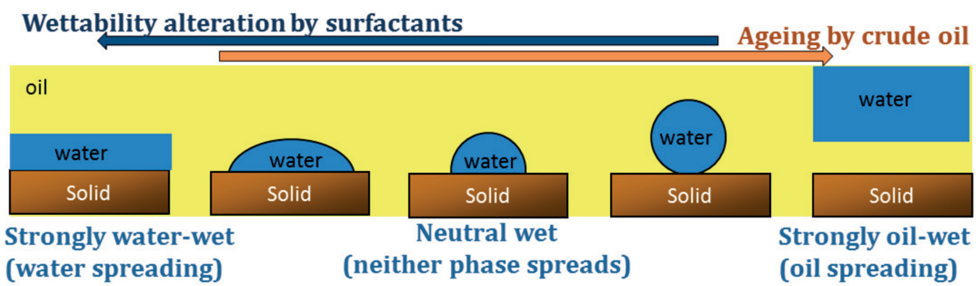

Figure 1: Wettability of a simple mineral surface from water-wet (left), through neutral-wet (middle) to oil-wet (right). Wettability can be altered from water-wet towards oil-wet by ageing in crude oil. Wettability reversal by surfactant (back towards water-wet) for EOR is an option to increase oil recovery by spontaneous imbibition.

Capillary continuity

Capillary continuity was originally investigated in conjunction with gravity drainage and believed to be prevailing in the vertical direction (Saidi *et al.* 1979, Horie *et al.* 1990, Labastie 1990, Stones *et al.* 1992), but may occur in any direction (Graue *et al.* 2000a, Graue *et al.* 2000b, Graue *et al.* 2001a, Graue *et al.* 2001b, Aspenes *et al.* 2002, Rangel-German *et al.* 2006, Aspenes *et al.* 2007). Capillary continuity between matrix blocks is vital to maintain a producing pressure gradient across several partly or completely isolated matrix blocks in a fractured reservoir, and improves oil recovery from gravity drainage and viscous displacement (Horie and Firoozabadi 1988, Labastie 1990, Stones *et al.* 1992).

Graue *et al.* (2000a, 2000b, 2001a, 2001b) and Aspenes *et al.* (2002, 2007) used MRI (Magnetic Resonance Imaging) to investigate fluid transport across vertical fractures during waterfloods. In strongly water-wet systems, block-by-block displacement of oil was observed: vertical fractures between matrix blocks filled hydraulically by the wetting phase, and were filled completely before water entered the next matrix block. At less water-wet conditions, water droplets formed on the fracture surface and grew into liquid *wetting phase bridges* that transported fluids across fractures up to 2.3 mm aperture, and into the next matrix block. A differential pressure was also transmitted across the open fractures through the liquid bridges, which reduced the impact of fractures on waterflooding and provided an additional viscous component to the flood. The viscous component compensated for the loss in oil recovery by capillary imbibition. No wetting phase bridges were observed in strongly water-wet fractures due to the strong water preference of the system, causing water to spread on the fracture surface rather than forming droplets. The proposed mechanism for fracture crossing is sketched in **Figure 2**.

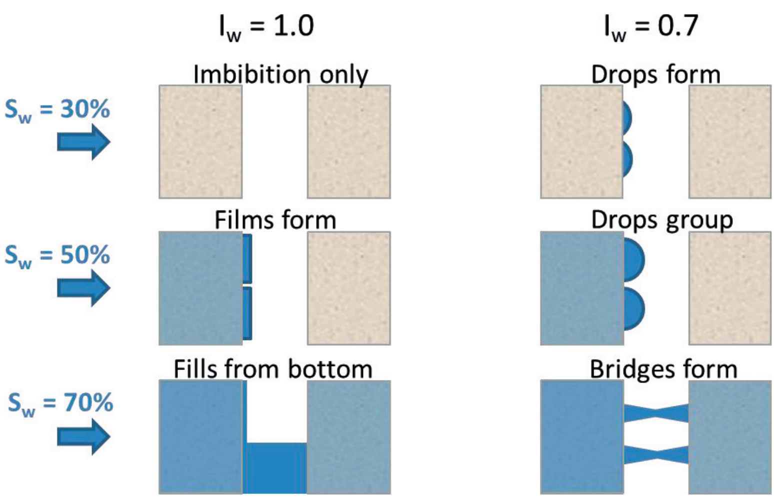

Figure 2: Proposed mechanism for fracture crossing, modified from Aspenes *et al.* (2007). For strongly water-wet systems (left), films form on the vertical fracture surface at water breakthrough, and the water migrates to the bottom of the fracture, displacing oil upwards. Water throughput to the second matrix block occurs when the fracture is completely filled by water in a hydraulic process. For less wetted systems (right), water drops form at the fracture surface at relatively low water saturations. As the water drops group and grow, they gradually form bridges that allow water to cross over into the next matrix block.

Recovery mechanisms

Gravity drainage

Oil recovery by gas-oil gravity drainage was first investigated in the 1940s (Cardwell and Parsons 1949) and can contribute to significant recoveries in fractured reservoirs with high vertical permeability (Firoozabadi 2000). Oil displacement by gravity drainage occur when gravity forces dominate over viscous and capillary forces (Hagoort 1980), and is therefore determined by the density difference between the gas in the fracture network and the matrix oil, and the effective matrix block height. In short matrix blocks, a capillary threshold pressure in the matrix will prevent entry of gas into the matrix by gravity forces. Gravity drainage is thus generally poor in highly fractured media without large continuous column heights, because of the large contrasts between matrix and fracture capillary pressure. The process can be significantly improved by establishing capillary contact between consecutive matrix blocks, thereby increasing the effective height of the matrix, or by reducing interfacial tension, thereby lowering the capillary contrast between the matrix and the fracture network. The latter can be achieved by gas injections above MMP (*minimum miscibility pressure*).

Spontaneous imbibition

Spontaneous imbibition is believed to be the governing mechanism for oil recovery in fractured reservoirs (Morrow and Mason 2001, Mason and Morrow 2013) and is particularly pronounced due to the lack of transition zones: the water-oil level has an advancing behaviour in the fracture system, and spontaneous imbibition into matrix blocks may exhibit both countercurrent (water surrounds entire matrix block) and co-current (matrix block partially covered in water) behaviour (Firoozabadi 2000). Significant oil recoveries may be achieved during waterflooding at water-wet conditions due to spontaneous imbibition, where displaced oil is produced into fractures or through consecutive matrix blocks in capillary contact with each other. The recovery of oil by spontaneous imbibition is governed by several factors, above all capillary pressure and wettability (Zhou *et al.* 2000). Initial water saturation (Viksund *et al.* 1998), interfacial tension between the fluid phases (Ma *et al.* 1997, Karimaie and Torseter 2007), matrix block size and surface area open to flow (Mattax and Kyte 1962) are also important factors.

1.2. Conventional recovery methods

Primary oil recovery by natural pressure depletion traditionally yield low oil recovery factors, below 15% of the oil originally in place (OOIP), although pressure in some reservoirs can be maintained for an extended period due to an expanding aquifer or gas cap. To improve oil recovery, secondary recovery efforts are often utilized, where fluids (water or gas) are injected to maintain reservoir pressure and displace hydrocarbons towards producing wells.

Waterflooding

Waterflooding is a common secondary oil recovery method, especially in regions where the injection fluid (water) is readily available, and waterflooding therefore is cost-effective. In non-fractured, porous rock, waterflood efficiency is largely determined by wettability (Anderson 1987b), which controls the post-waterflood residual oil saturation (Morrow

1990). Fractional flow calculations show that fluid displacement is viscously driven (Rapoport and Leas 1953). In fractured reservoirs, highly conductive fractures separate the matrix blocks and limit the influence of viscous forces (Bourbiaux 2009), hence the success of a waterflood is controlled by capillary forces and gravity (Anderson 1987b). Oil recovery by waterflooding in fractured reservoirs depends strongly on spontaneous capillary imbibition and is thus dependent on wettability.

Waterflood oil recovery from fractured reservoirs has in some cases been unexpectedly high, as observed in the Ekofisk field on the Norwegian continental shelf (NCS): waterflooding was initially initiated for pressure support, due to significant seabed subsidence after years of production by pressure depletion (Hermansen *et al.* 1997, 2000). Significant increased oil recovery followed due to favorable wettability (spontaneous imbibition) and weakening of the chalk reservoir rock (compaction drive) combined. **Figure 3** shows the field oil rate response to waterflooding.

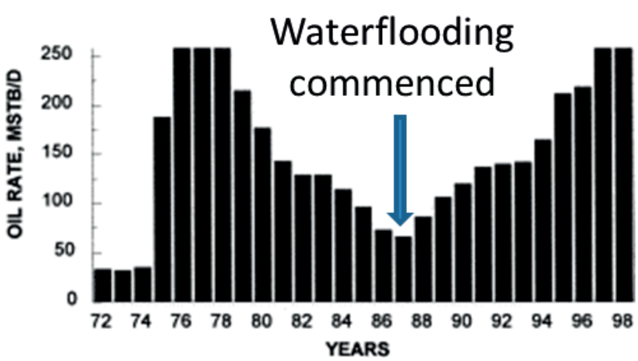

Figure 3: Response in oil production rate from waterflooding in the Ekofisk field, modified from Hermansen *et al.* (2000).

At oil-wet or neutral wetting conditions, the capillary contribution to flow is low, and little to no oil is produced by spontaneous imbibition. The contribution from viscous displacement mechanisms is also low due to fractures, hence poor volumetric sweep and low oil recovery is frequently observed during waterflooding, and the majority of water flows through fractures only. Large volumes of oil are left behind, trapped in matrix blocks surrounded by fractures, and the potential for enhanced oil recovery (EOR) is high. Conformance control decrease the influence of fractures on flow, and enable chase fluids to enter the matrix blocks at increased viscous pressure gradients. Reduction of fracture permeability by e.g. polymer gel can improve chase waterflood efficiency in fractured reservoirs.

Gas flooding

Gas flooding is an alternative secondary oil recovery method, where produced hydrocarbon gas is re-injected for pressure support, either into the same reservoir from which it was produced, or neighboring reservoirs. Gas flooding can also be implemented as an EOR method for miscibly displacing matrix oil, using CO_2, hydrocarbon-miscible gas e.g. C_2, C_3, C_4, flue gas and nitrogen, further discussed in **Chapter 2** and **Chapter 3**.

Gas is either immiscible or miscible with the reservoir oil during displacement. Immiscible gas floods in fractured media are not efficient, due to segregation of liberated gas to the top of the fracture system. Miscible gas displacements significantly decrease the interfacial tension between oil and gas and ultimately reduce capillary contrast between the matrix and fracture to zero. Injected fluids are therefore able to flood the matrix in addition to the fracture network, thus improving local displacement efficiency and oil recovery by gravity drainage and viscous flooding (Orr and Pande 1989, Firoozabadi 2000). The high local displacement efficiency may, however, be offset by low macroscopic sweep efficiency due to fluid channeling (Bank *et al.* 2007). Permeability heterogeneity, therein fractures, contribute to severe fluid channeling during miscible gas floods, and is aggravated by high adverse mobility ratios and large density contrast between the injected gas and reservoir oil (Stalkup 1984). Crossflow between matrix and fractures has been verified during miscible displacements (Firoozabadi 1994, Tan and Firoozabadi 1995, Dindoruk and Firoozabadi 1996), and causes fluid mixing and significant amounts of two-phase flow. The local displacement efficiency is reduced due to crossflow. Macroscopic sweep efficiency and overall recovery of oil are, however, improved, due to a reduction in fluid mobility, which reduce channeling caused by permeability heterogeneity and gravity override (Orr and Pande 1989, Pande 1992). The mobility during gas floods may also be improved by mixing a surfactant into the gas phase, to generate foam. Foam injections add a viscous component to the gas flood and reduce the impact of fractures on fluid flow.

2. Enhanced oil recovery (EOR) in fractured reservoirs

In many fractured reservoirs, secondary recovery efforts do not yield sufficient increased oil recovery, and remaining oil volumes can be considerable. *Enhanced oil recovery (EOR)* methods can be implemented to mobilize the oil and displace it towards production wells. EOR refer to injection of fluids or chemicals that are not naturally present in the reservoir (e.g. surfactants, polymers, acids, clean gases) to achieve a higher or faster oil recovery. Oil recovery may also be improved by drilling of additional (e.g. horizontal) wells to improve the flow pattern in the reservoir, often termed Improved Oil Recovery (IOR).

2.1. Conformance control

The main challenge in EOR operations is enabling the injected fluids to contact the hydrocarbons stored in the matrix. In fractured reservoirs with limited spontaneous imbibition, fracture/matrix capillary contrasts cause injected fluids to flow in the high permeable fracture network during EOR operations, while oil remain trapped in the matrix. Fracture channeling is defined as a c*onformance problem*, alongside viscous fingering due to poor mobility control, matrix channeling in high permeable layers, water coning and well problems (Flores *et al.* 2008, Sydansk and Romero-Zerôn 2011). Conformance problems cause non-uniform displacement fronts, which results in early breakthrough of injected fluids and low production oil cuts. Channeling or fingering of fluids brought about many disappointing EOR field tests in the 1970s and 1980s, because the injected EOR fluids and gases were inhibited from contacting significant portions of the reservoir (Sydansk and Romero-Zerôn 2011).

Improved conformance in a fractured reservoir can be achieved by 1) reducing fracture permeability (Graue *et al.* 2002), and/or 2) establishing a viscous pressure component in the matrix, e.g. during chase floods after fracture permeability reduction, or by increasing the apparent viscosity of the injected drive fluid. According to Sydansk and Romero-Zerôn (2011), successful conformance control may contribute to:

- Improved sweep efficiency.
- Increased and incremental oil recovery.
- Accelerated oil recovery rates.
- Reduced oil recovery expenses, because recycling of drive fluids is reduced, which also reduce the associated lifting, handling, treatment, environmental-related, and disposal costs.
- Reduced environmental liabilities and increased environmental benefits (e.g. produce less saline, heavy-metal-containing, and/or H_2S-containing water).

Conformance control by gel

Reduction of flow in fractures or high permeability zones by placement of gels have been reported (Seright and Martin 1991, Seright and Liang 1994, Seright 1995, 1997, Tweidt *et al.* 1997, Seright *et al.* 1998, Portwood 1999, Sydansk and Southwell 2000, Seright *et al.* 2001, Portwood 2005, Rousseau *et al.* 2005, Alhajeri *et al.* 2006, Willhite and Pancake 2008, Spildo *et al.* 2009, Stavland *et al.* 2011). Gels are most often used for fracture blocking purposes. Fracture permeability reduction provided by the gel enable increased differential pressure gradients across matrix blocks during subsequent floods. Injected chase fluids or gases can thus be diverted to areas that have not previously been swept. **Figure 4** illustrates fracture channeling during oil recovery: a known conformance problem which has been successfully treated through *polymer gel* applications.

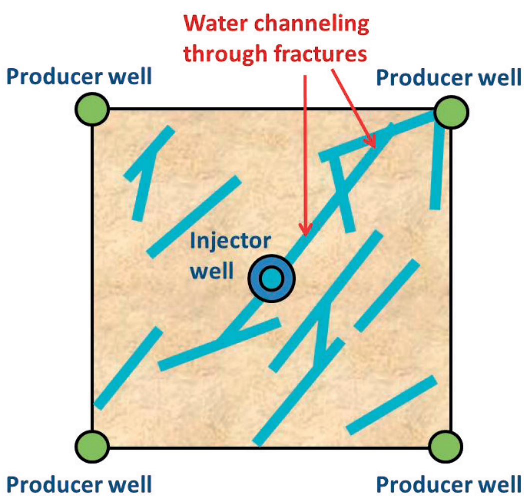

Figure 4: Fracture channeling during waterflooding, modified from (Sydansk and Romero-Zerôn 2011)

Foams for conformance improvement during gas floods

Mixing of surfactant and gas to generate *foam* reduce the mobility of the gas phase by making it discontinuous: during mixing, the gas is dispersed into bubbles and separated by thin liquid films (*lamellae*). Foams have both 1) a viscous-enhancement component, and 2) a permeability-reducing component that can be exploited for conformance improvement (Sydansk and Romero-Zerôn 2011). Foam injection for conformance control have been studied both experimentally and numerically (see e.g. (Hirasaki and Lawson 1985, Falls *et al.* 1988, Kovscek *et al.* 1995, Rossen 1995, Geiger-Boschung *et al.* 2009)), and shown to be applicable in heterogeneous (Bertin *et al.* 1999b) as well as fractured (Haugen *et al.* 2012) reservoirs. In heterogeneous reservoirs, foam will generate in high permeable zones first and divert flow into zones of lower permeability. After entry into the low permeable zone, the foam front moves with equal velocities in zones of both high and low permeability when there is capillary communication and crossflow between these (Bertin *et al.* 1999a). Foam also greatly enhances the sweep in fractures (Kovscek *et al.* 1995, Yan *et al.* 2006, Pancharoen *et al.* 2012) and reduce the effect of longitudinal fractures on flow (Panahi 2004). Application of foams for enhanced oil recovery (EOR) during gas floods is a promising technique at both immiscible and miscible conditions and using a variety of gases.

2.2. Polymer gel

In naturally fractured reservoirs, successful conformance control is required in the far-wellbore region as well as in the near-well area. Gel technology represents an opportunity to treat all sections of a reservoir. Several thousand different gel systems exist, and an overview of some gels, used for the purpose of oilfield conformance control, is given in **Table 2**. Polymer gels have emerged to become the most widely applied conformance-improvement gel technology (Sydansk and Romero-Zerôn 2011), and are high flowing, yet rigid, blocking agents that may be placed deep in the reservoir. The experimental work using polymer gel in this thesis focus on *Chromium (III)-Carboxylate/Acrylamide-Polymer (CC/AP) Gels,* specifically Cr(III)Acetate-HPAM gel. This gel system, first investigated by Robert Sydansk (Sydansk and Argabright 1987, Sydansk 1988, 1988, 1990), has been applied world-wide as a conformance-improvement treatment, and is a field scale proven technique. Its robust gel chemistry has been widely studied and is well documented.

Cr(III)Acetate-HPAM gel

Cr(III)Acetate-HPAM polymer gel is a semi-solid material, formed through a reaction between a polymer solution and cross-linker agent, as shown in **Figure 5**. Water is first mixed with a polymer to form a polymer solution (**Figure 5**, left). The polymer concentrations used for oilfield conformance improvement vary from 1500-100000 ppm but is typically in the range of 3000-12000 ppm. When a cross-linker agent is added to the polymer solution (**Figure 5**, middle), a *gelant* is formed. Gelant has low viscosity and small particles, which enable it to flow through rock matrix as well as fractures (Seright *et al.* 2003). Subjecting gelant to an elevated temperature over time changes the solution properties and a rigid gel is formed (**Figure 5**, right). The process where gelant transforms to gel is called “cross-linking” or “gelation”.

Table 2: Gel technologies for use in oilfield conformance control.

Gel technologies used for oilfield conformance control		
Inorganic gels		
Silicate based		
Aluminum based		
Hydroxides of selected divalent and trivalent cations		
Organic polymer gels		
Biopolymers	Xanthan	Organic cross-linking agents
Synthetic polymers*	Acrylamide-based polymers*	Aldehydes
		-Phenol-formaldehyde and derivatives
		Polyethyleneimine
		Inorganic (metal) cross-linkers
		Al(III)
		Zr(IV)
		Cr based
		-Cr(III) w. inorganic anions
		-Cr(VI) redox
		-Cr(III) w. organic carboxylate complex ions*
Monomers	Acrylamide monomer	
	Acrylate monomer	
	Phenolics	
Lignosulfonate gels		
Preformed particle gels (PPG)	Swelling organic-polymer "macroparticle" gels	
Microgels		
Microgels with narrow particle-size distribution		
CDGs	Aluminum citrate cross linked	
	Chromic-triacetate cross linked	
Delayed "popping"/swelling microgels (Bright Water™)		
Mixed silicate and acrylamide-polymer gels		

***The polymer gel highlighted in green letters is the most commonly used gel system for oilfield conformance treatments, and was also used in the experimental section of this thesis.**

The rigidity of the gel can be controlled by varying the chemical concentrations, and can range from highly flowing to rubbery (Sydansk 1988). The concentrations of polymer and cross-linker are usually low and the solvent (commonly water, although oil-based gels also exist) is the main ingredient in the polymer gel. Most polymer gels have an initial water content of 95 to 99.7% (Sydansk and Southwell 2000).

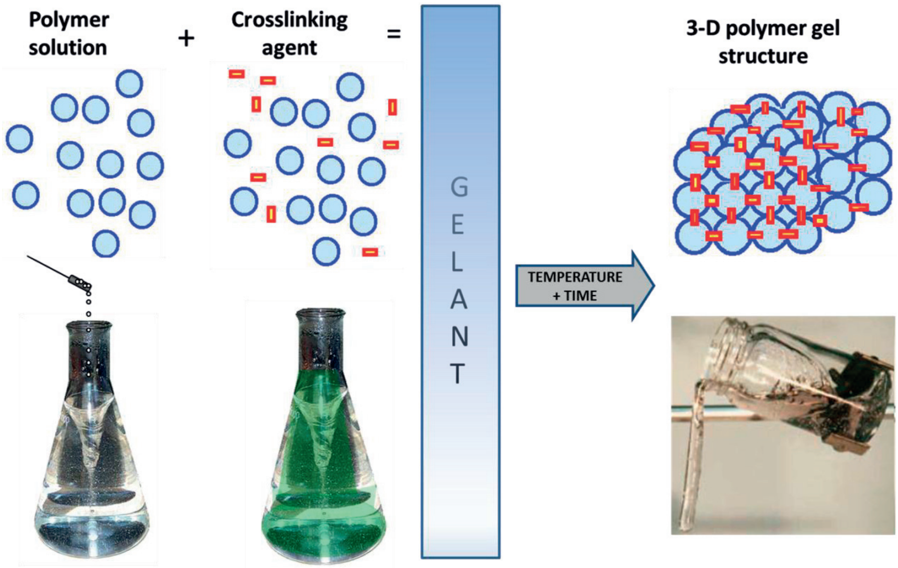

Figure 5: A polymer solution (left) forms a gelant when a cross-linker agent is added (middle). When the gelant is subjected to a higher temperature over time, a rigid gel is formed (right).

Gel placement in fractures

Polymer gel can be placed in fractures as immature gel (gelant) or as pre-formed, mature gel. Immature gel may flow through porous rock as well as fractures during placement, and relatively low pressure gradients are required for extrusion (Seright *et al.* 2003). Immature gel is used in both high permeability layers and fracture blocking applications. The cross-linking process occur *in-situ* at reservoir conditions. Immature gel applications may experience a series of drawbacks (Bai *et al.* 2008), including:

- *Lack of gelation time control*. Contact with reservoir fluids or rock may alter the composition of the gelant and interfere with gelation. Delayed or incomplete cross-linking due to dilution by formation water, chromium retention by precipitation/adsorption, polymer retention and pH changes have been observed (Seright 1992, Seright and Martin 1992, 1993, Stavland and Nilsson 1995, Zou *et al.* 2000, Ganguly *et al.* 2002, Jin *et al.* 2003, Chen *et al.* 2010).

- *Gelant intrusion into zones not previously flooded by water*. The gelant viscosity increase during gelation, which can enable the gelant to enter into zones in the reservoir that have not been waterflooded, e.g. potentially productive zones of high oil saturation. If gel forms in these zones, they may be severely damaged. It has been suggested that capillary forces in oil-wet formations will prevent gelant invasion into zones of high oil saturation, leaving oil bearing zones unharmed during gelant injection, but this was contradicted by Liang *et al.* (1993): they found that gelant may penetrate into all open zones during placement in production wells, including zones of high oil saturation.

Mature gel is inhibited from passing through pore throats due to its long chained structure, and cannot flow through porous rock. The chemical treatment is therefore limited to open fractures during injection (Seright 2001a), and productive oil zones will not be harmed. The fracture volume typically constitutes ~1% of the total reservoir pore volume, and selective chemical treatment of fractures limits the need for chemicals. Mature gel has little sensitivity to physiochemical conditions in reservoirs (Zhang and Bai 2011), and is less prone to gravity segregation in fractures compared to gelant (Seright 1995). The drawbacks of mature gel injection are linked to its behaviour during injection:

- *High pressure gradients are required for gel injection*. The effective viscosity of formed gel in fractures is typically a factor 10^3 to 10^6 higher than for gelant, and mature gel injection thus require higher injection pressures. Mature gel enters, and extrudes through, a fracture at an elevated pressure gradient, controlled by the fracture aperture. The pressure gradient is insensitive to the flow rate and progressive plugging does not occur during gel extrusion through fractures, i.e. the pressure does not increase (Seright 2001b, 2003a).
- *The gel treatment is limited to open fractures*. Gels do not enter porous media or narrow fractures (below 0.052 mm aperture) after they have matured (Seright 2004), which may limit the gel treatment to near wellbore regions or fractured wells.

In most field applications, gelant is mixed at a surface facility and immediately injected through the wellbore. Treatment size and injection time varies, but will in most cases surpass the inherent short gelation time of the Cr(III)-HPAM gel system, of approximately 5 hours at 41^{o}C. The most successful gel treatments of naturally fractured reservoirs required injection of large volumes of gel, and injection times far exceeding the gelation time (Sydansk and Moore 1992, Borling 1994, Hild and Wackowski 1999a). Thus, mature gel extrude through fractures during most of the placement process. To achieve in-depth gel treatments during mature gel injection, a continuous fracture system is required, through which gel can extrude. A second option to achieve far wellbore treatments by gel is to prolong the gelation time, thus extending the distance an immature gel is able to propagate prior to gelation. Cordova *et al.* (2008) successfully extended the gelation time of the Cr(III)-

HPAM gel system, by using Polyelectrolyte complex nanoparticles to entrap and control the release of Cr(III).

Gel resistance to washout during chase floods

After polymer gel placement in fractured systems, chase fluids are injected to displace oil from matrix blocks. The behaviour of the gel during chase floods largely determines the success of the conformance treatment. Polymer gel resistance to washout from fractures may be described by a *rupture pressure*, which is the highest pressure a gel in a fracture can resist before fluid transport through the gel occurs. Rupture pressures have been measured experimentally after placement of both immature (Ganguly *et al.* 2002, Wilton and Asghari 2007) and mature gel (Seright 2003b) in fractured systems.

Ganguly *et al*. (2002) injected gelant through fractured cores and slabs, and allowed the cross-linking process to occur *in-situ*. Gelant was in some experiments injected into both the core matrix and the fracture, where gelant intrusion to the matrix was controlled by adjusting the injected volume. Gel resistance to washout was tested during chase waterfloods, by measuring the rupture pressure, and was observed to be higher when gel had formed in the fracture and adjacent matrix. When gelant was placed in the fracture alone, without saturating the adjacent matrix, gel did not form, presumably due to diffusion of chromium through the porous rock. Ganguly *et al.* argued that gelant, when injected through both the fracture and surrounding rock matrix, formed a zone of homogeneous concentrated gel during cross-linking. This created a gripping effect between gel in the fracture and gel in the matrix that increased its overall pressure resistance. Wilton and Asghari (2007) achieved *in-situ* cross-linking without gelant leakoff to the matrix by pre-flushing the core with chromium solution, or by placing gelant with an increased amount of chromium in the fracture (chromium overload).

Seright *et al*. (1998, 2003) and Seright (1995, 1999, 2003a) showed that mature gel dehydrated during propagation through a fracture. The dehydration process was termed *leakoff*, and occurred due to the pressure difference between the fracture and matrix during gel injection. During gel propagation with leakoff, solvent left the gel and progressed through the core matrix, leaving a gel of higher concentration and rigidity in the fracture. The injected, mature gel flowed through the dehydrated, concentrated gel in designated flow channels (*wormholes*). As leakoff progressed, wormhole size diminished and the polymer concentration in the dehydrated gel layer increased. This concentration was a direct measure of the gel ability to divert chase water, and the pressure resistance of the gel increased with increasing polymer concentrations in the dehydrated gel layer. The non-dehydrated gel contained in the wormholes could be readily mobilized, and was the weakest link during subsequent floods. Seright (2003b) injected mature gel into open fractures and recorded solvent leakoff. Rupture pressures were measured during water or oil chase floods. He found that the pressure required to mobilize gel in the wormholes during chase floods was comparable to the pressure attained during gel placement, which was largely controlled by the fracture aperture.

Previous work could not be used to directly compare the two gel placement methods, due to variations in fracture aperture, core material and chemical composition, as well as the general experimental setups. In **Paper 1**, gel rupture pressure and blocking capacity after rupture was therefore investigated experimentally in similar systems to accurately evaluate and compare the methods. Immature gel and mature gel were placed in fractured core plugs for conformance control, and gel resistance to washout was measured during water chase floods. Unified core properties, fracture dimensions, assembly methods and experimental conditions ensured direct comparability of the two placement methods.

Gel stability after placement in fractures

After placement in a fracture, gel may undergo processes that reduce its pressure resistance, e.g. syneresis and dehydration. Syneresis is caused by chemical reactions over time, and believed to be largely controlled by an increasing cross-link density, which causes the gel volume to shrink and consequently expel water (Bryant *et al.* 1996, Nguyen *et al.* 2000, Vossoughi 2000, Romero-Zeron *et al.* 2008). Nguyen *et al.* (2004) reported that syneresis caused a maximum decrease in gel volume of 1.2% during the course of four months. Dehydration is characterized by a reduction of the gel volume due to the expulsion of solvent from the gel, and has previously been observed during fluid flow through micro models and sand packs containing gel (Dawe and Zhang 1994, Al-Sharji *et al.* 1999, Nguyen *et al.* 2004), and through bulk volumes of gel (Krishnan *et al.* 2000). These authors suggested that imposing a pressure gradient on the gel after placement may displace solvent from the gel volume and cause dehydration. The reduction in the gel volume due to dehydration totaled 50-70% in their works.

Although gel behaviour after placement in fractures has been widely studied, it is rarely discussed in conjunction with the properties of an adjacent, oil-saturated matrix. Relative permeability and capillary pressure significantly influence the flow of water in fractured media, and may thus also influence gel behaviour during and after placement, considering that aqueous gels consist mainly of water. **Paper 2** presents a series of experiments where the influence of capillary forces on gel behaviour after placement was evaluated. Strongly water-wet core plugs were saturated by oil and placed in contact with bulk volumes of mature gel. Shortly after contact, capillary forces in the core matrix contributed to spontaneous imbibition of solvent from the gel, i.e. gel dehydration was experimentally verified to occur due to capillary spontaneous imbibition, without imposing a pressure gradient on the system. The gel volume severely decreased due to the extraction of solvent by spontaneous imbibition.

2.3. Carbon dioxide- CO_2

CO_2 flooding for enhanced oil recovery has been commercially applied since the 1970s, when it was first used in Texas (Lambert *et al.* 1996). The use of CO_2 for EOR is rapidly increasing; in 2012, CO_2 EOR contributed to approximately 5% of the USA domestic oil production, and is likely to double by 2020 (Enick and Olsen 2012, Kuuskraa and Wallace 2014).

Recent focus on reducing greenhouse gas emissions has at the same time led to increased efforts within Carbon Capture Utilization and Storage (CCUS). Storage and utilization of CO_2 in mature oil fields is possible, and research emphasizing CO_2 injection for safe long-term storage, and simultaneous CO_2 EOR is currently of great interest, world-wide. CO_2 for EOR has not yet been applied on the Norwegian Continental Shelf (NCS). However, several new carbon capture projects are being planned (Norwegian Ministry of Petroleum and Energy 2011), and recent published data by the Norwegian Petroleum Directorate (Halland *et al.* 2014) describe the potential for using CO_2 in full-scale EOR projects in the North Sea. CO_2 flooding was investigated as an oil recovery method in fractured carbonates in this thesis.

CO_2 floods are immiscible or miscible depending on the reservoir conditions (pressure, temperature and crude oil composition), and are also applicable for WAG (Water Alternating Gas) operations. **Figure 6** shows basic information on the change of CO_2 viscosity and density as functions of temperature and pressure.

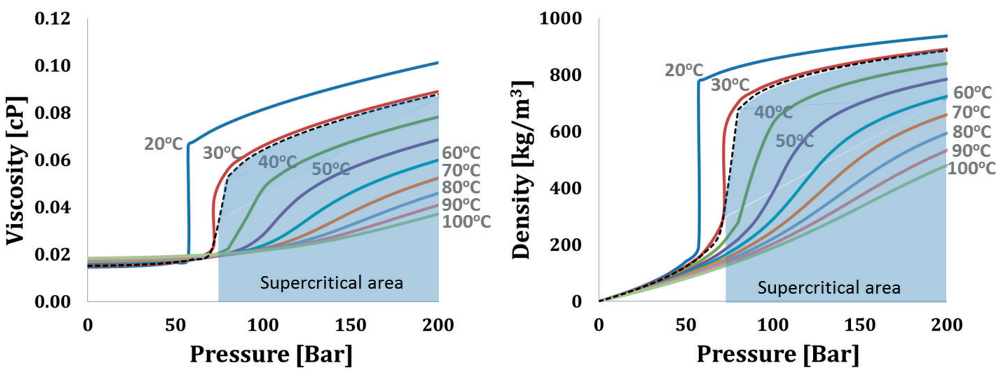

Figure 6: Change in viscosity (left) and density (right) as functions of pressure and temperature. The black, dotted line represents a temperature of 31.1°C, which at 73.8bar constitute the critical point, where CO_2 transform from gas or liquid phase to supercritical phase. Data generated from Lemmon *et al.* (2014).

Miscible CO_2 flooding

The primary objective of a miscible CO_2-EOR flood is to reduce post-waterflood residual oil saturation (Bank *et al.* 2007). In 2010, 153 miscible CO_2 floods were executed, of which 139 were in the USA (Al-adasani *et al.* 2012). Because of its properties at typical reservoir conditions, CO_2 is favorable over other gases for use in EOR: CO_2 tends to be miscible with reservoir oil at lower reservoir pressures (Holm 1986), and maintain a high viscosity at higher pressures and temperatures than other miscible gases, e.g. methane (Lambert *et al.* 1996).

Miscibility is defined as a "the ability of two or more substances to form a single homogeneous phase when mixed in all proportions" (Holm 1986), and may occur when gas is injected to displace oil above the minimum miscibility pressure (MMP). MMP vary with e.g. injected gas composition, reservoir temperature and crude oil composition (Yuan *et al.* 2005). In fractured reservoirs, miscibility between reservoir crude oil and CO_2 remove capillary contrasts and promote viscous crossflow between matrix and fractures, which enable the recovery of capillary trapped oil. Light hydrocarbons vaporize more easily into the gas phase than heavy hydrocarbon components (Holm and Josendal 1974, 1982, Silva and Orr 1987, Hagedorn and Orr 1994), and CO_2 can obtain first contact miscibility with single-component light oils, e.g. n-Decane. First contact miscible fluids instantly mix and form one phase at contact, when mixed at any ratio. Crude oils are complex and have several components, hence CO_2 is not first-contact miscible with most reservoir oils (Martin and Taber 1992, Hemmati-Sarapardeh *et al.* 2014). Multi-contact miscibility can, however, develop through a mix of condensing (CO_2 condense into the oil) and vaporizing (light oil components vaporize into the CO_2) gas drives. After a series of mass transfers between reservoir oil and CO_2, the interfacial tension between the two diminish and they appear as one phase (Ghomian *et al.* 2008). In **Paper 3**, miscible CO_2 floods for EOR were investigated experimentally at reservoir conditions using different oils. CO_2 floods were initiated in both fractured and non-fractured chalk, where n-Decane and North Sea crude oil constituted the oil phases. Important recovery mechanisms in miscible CO_2 flooding are (Bank *et al.* 2007):

- Oil swelling and decreased oil viscosity, which both occur when reservoir oil and CO_2 mix.
- Extraction of lighter hydrocarbons into the gas phase.
- An additional viscous pressure in the drive fluid.

The viscous pressure is often absent during gas floods in fractured systems. Dispersion of fluids around the displacement front is, however, of significant importance. Dispersion by convection (mechanical mixing) is the prevailing mechanism in the fracture network, while dispersion by diffusion will influence matrix flow. Dispersion may occur due to differences in concentration (molecular diffusion), temperature (thermal diffusion) or pressure (pressure diffusion) between two areas. Molecular diffusion is especially important in miscible gas flooding in fractured reservoirs (da Silva and Belery 1989, Hu *et al.* 1991, Jamili *et al.* 2011), and Haugen and Firoozabadi (2006) suggested that the mechanism may contribute to up to

25% of the oil recovery. The contribution from molecular diffusion to oil recovery is increased when the flow rate in the matrix is low or when the contact area between injected gas and reservoir oil is high, e.g. in fractured reservoirs.

The presence of water in the pore space may influence oil recovery by CO_2. Good contact between the injected gas and matrix oil is important to achieve oil recovery by diffusion, but may be inhibited by *water shielding*, where water films prevent contact and mixing between CO_2 and reservoir oil. CO_2 is highly soluble in water, which can aid diffusion through thin water films to recover oil during tertiary recovery applications (Martin and Taber 1992). High water saturations at the onset of CO_2 flooding may, however, inhibit displacement of oil from dead end (*dendritic*) pores or clusters, leaving higher residual oil saturations (Campbell and Orr 1985). Water shielding and the effects on CO_2 EOR were investigated in **Paper 3** during tertiary CO_2 floods at different wetting conditions. Wettability controls the distribution of fluids in the pore space and may therefore influence the location and presence of water films, and hence the efficiency of CO_2 oil recovery.

CO_2-foam

High microscopic sweep efficiency is often achieved in miscible CO_2 floods. Macroscopic sweep efficiency may, however, be low due to the high mobility of the gas phase, which promotes gravity segregation and fluid channeling through fractures. CO_2 mobility may be reduced by mixing of surfactant solution and gas to generate foam. Foam increase the apparent viscosity of the gas phase and provide a viscous pressure to the oil recovery process. An important factor in foam flooding is *foam strength*, which comprise the apparent viscosity, texture and stability of the foam in fractures and porous media (David and Marsden 1969). Foam strength depends e.g. on the amount of gas dispersed in the foam: *the gas fraction* (f_g) commonly range from f_g = 0.9 ("dry" foams) to f_g = 0.5 ("wet" foams). Strong foams are recognized by their ability to move trains of dispersed gas bubbles in a system and improve mobility more than "weak" foams, where gas and surfactant solution may flow more or less separately, albeit at higher injection pressure gradients (Li *et al.* 2006).

Foam can, like gas floods, be either miscible or immiscible with the reservoir oil. Immiscible foam floods can be advantageous for enhanced oil recovery, compared to immiscible pure gas, because they provide an additional viscous pressure gradient to the gas flood. Haugen *et al.* (2012) studied immiscible N_2-foam injections in fractured, oil-wet carbonates, where gas floods prior to conformance control by foam contributed below 10% oil recovery. By injecting pre-generated N_2-foam, oil recovery was improved, and up to 80%OOIP oil recovery was observed. The high recovery was, however, reached after more than 100PV of foam was injected, and indicated that the oil was displaced by surfactant solution only. In **Paper 4**, N_2- and CO_2-foam was injected into fractured oil-wet core plugs at immiscible and miscible conditions to improve oil recovery. A medical CT scanner was used to investigate flow mechanisms and gas flow patterns during immiscible foam injections at different wettability conditions. We found that gas did, in fact, not enter the matrix at oil-wet conditions, and oil recovery was therefore attributed to the surfactant.

Surfactants can be used independently to promote oil recovery by wettability alteration and spontaneous imbibition, and has shown promising results in the lab with up to 70%OOIP recovery (Austad and Milter 1997, Standnes *et al.* 2002). A major challenge in fractured, oil-wet reservoirs, without significant viscous contributions to fluid flow, is enabling the surfactant to contact matrix oil. Foam implementations may, in addition to improving gas flooding, provide surfactant entry to the matrix by a viscous component. Zuta *et al.* (2009) and Zuta and Fjelde (2010) have shown promising results during CO_2-foam injections in fractured chalk. Foam blocked fractures and high permeable regions and enabled CO_2 to diffuse and flow into the low permeable matrix. The surfactant solution flowing in the fracture system also eventually entered the matrix and recovered oil by wettability alteration and interfacial tension reduction.

2.4. Integrated EOR- an opportunity

Integrated EOR (IEOR) (see e.g. Haugen *et al.* (2010a), Fernø *et al.* (2012)) represents a valuable opportunity in EOR operations: combining several EOR methods may improve both the microscopic and macroscopic sweep efficiencies while limiting the need for expensive chemicals. An important goal is to combine methods that optimize conformance control both in-depth and in the near-well area, and thus provide efficient IEOR for fractured reservoirs.

Fractures often dominate fluid flow in a reservoir, and injected fluids channel through the high permeable fracture network instead of displacing oil from the matrix. Fracture channeling, although being a major challenge in many single fluid EOR processes, can also be an advantage: using expensive chemicals to treat the small fracture volume (typically ~1% of the total pore volume) may improve the performance of less expensive fluids in a multi-step integrated EOR approach. Consider the use of surfactants in a fractured reservoir: because the surfactant solution channel through fractures and contact very little of the matrix oil, injection of significant surfactant volumes may not be economical. Injection of smaller surfactant volumes to treat the fracture surfaces may, however, have a significant effect and be cost-effective. Altering fracture surface wettability towards neutral-wet or less wetting conditions may aid the establishment of capillary continuity by the formation of wetting phase bridges, and thus improve oil recovery from viscous displacement across consecutive matrix blocks during injection of chase fluids. Gel placement to improve conformance by fracture permeability reduction is another example: mature gel will not intrude into porous rock, and only propagate through open fractures. The next step in the integrated EOR process can then be a cost-effective traditional waterflood, a pure gas or foam chase flood, or injection of other EOR chemicals to flood the matrix. The Cr(III)-Acetate-HPAM gel system has a robust chemistry and is insensitive to most oilfield and reservoir interferences. It is also applicable over a broad pH range. These factors combined render the gel system applicable to the acidic conditions associated with CO_2 flooding, where most established oilfield polymer gels are not (Martin and Kovarik 1987, Hild and Wackowski 1999b).

Large volumes of chemicals have been used to attain high recovery factors in many previous EOR studies, i.e. the rates of oil recovery were low. One example is Haugen et al. (2012), who achieved 80% oil recovery, but only after several tens of foam pore volumes had been injected. In many foam applications (see e.g. **Paper 4**) strong foam has to be generated in the fracture before fluids enter and flood the matrix. Reducing fracture conductivity prior to gas or foam injection could enable fluids to enter the matrix at fewer pore volumes injected. **Paper 5** presents an integrated EOR approach, where reduction of fracture permeability by polymer gel was the first step. Mature gel was injected, which ensured treatment of the fracture volume only and reduced the need for chemicals. Subsequent water, surfactant and CO_2-foam chase floods were implemented to displace oil. The experimental steps were monitored by CT and MRI. *In-situ* imaging provided information on local fluid distributions during chase floods and enabled qualitative analysis of the shape of the displacement front.

3. Experimental work and results

Fluid flow through porous media is studied on different scales, ranging from pore scale to field scale (**Figure 7**). Experiments on pore, core and block scale enable selective investigations of single parameters, and their effect on fluid flow. Accurate experimental results are important to understand basic recovery mechanisms, which are needed to describe the complexity of fluid flow at field scale. The experiments in this thesis were performed on core scale. The core plugs were of 1.5'' and 2'' diameters and core lengths varied from 5 to 12 cm. Four outcrop core materials were used and are described below.

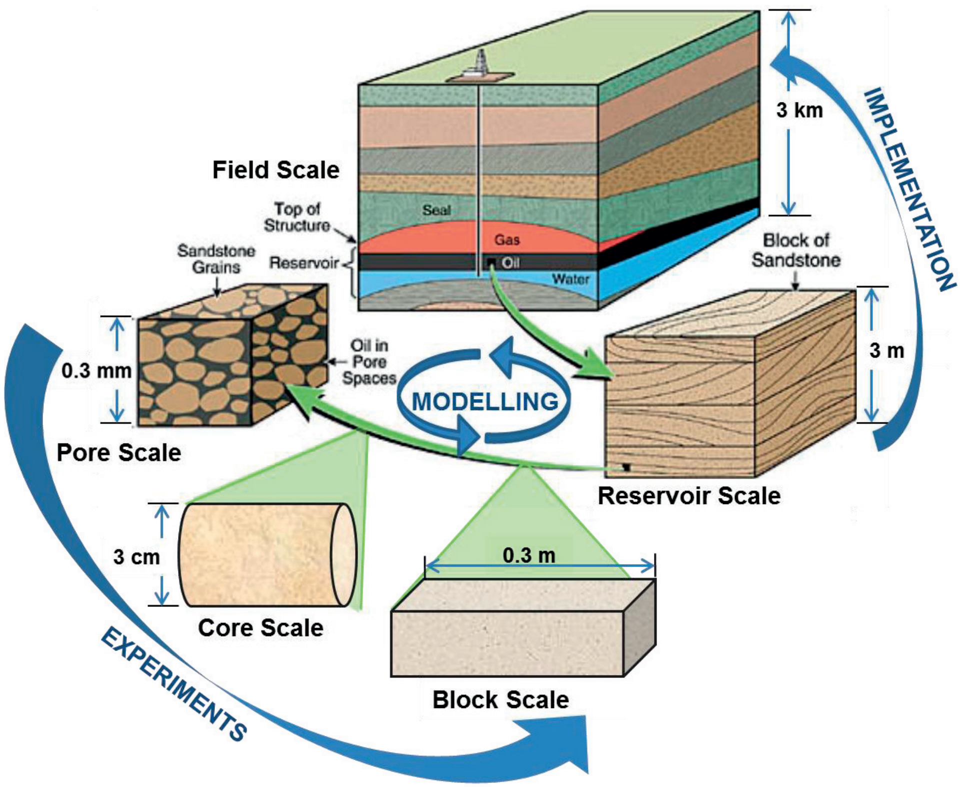

Figure 7: Different scales involved in hydrocarbon research. Parts of the figure was modified from Zitha *et al.* (2011)

- *Portland chalk* from the Portland cement factory in Aalborg, Denmark, also called Rørdal chalk. The rock formation is of Maastrichtian age and consists mainly of coccolith deposits. The composition is to a large extent calcite (99%) with some quartz (1%). Absolute permeability and porosity ranges from 1 to 10 mD, and 43 to

48%, respectively. The Portland chalk material is fairly homogeneous and was previously described by Ekdale and Bromley (1993) and Hjuler (2007).

- *Edwards limestone* from west Texas, USA. The limestone core material is non-uniform with permeability to water ranging from 3 to 28 mD and porosity values from 16 to 24%. Mercury injections, thin section images and NMR T2 relaxation experiments were used to characterize this rock type, identifying vugs, microporosity and trimodal pore sizes (Tie 2006, Johannesen 2008).
- *Bentheimer sandstone* from the Gildehausen quarry near Bentheim in Germany. A homogeneous sandstone in terms of porosity (23%) and permeability (1200 mD), with a composition of 95% quartz, 3% kaolinite, and 2% orthoclase (Schutjens *et al.* 1995, Klein and Reuschle 2003).
- *Berea sandstone* from Ohio, USA. A Mississippi terrestrial sandstone with homogeneous permeability of 500 mD and approximately 25% porosity, which consist predominantly of quartz (85-90%) and feldspar (3-6%), with small amounts of dolomite (1-2%), clays (6-8%) and iron sulphide (trace amounts) (widely used, see e.g. Churcher *et al.* (1991)).

Cylindrical core plugs were drilled from larger rock slabs, and cut to length. The surfaces were cleaned using fresh water and the core plugs were thereafter stored at 80^{o}C for several days until completely dry. The core plugs were either saturated by brine and/or mineral oil and used in the strongly water-wet state, or aged by crude oil towards less water-wet or oil-wet conditions. Wettability alteration was performed using a dynamic ageing method based on continuous flushing of crude oil through the core plug at an elevated temperature, extensively described and referenced within Johannesen (2008) and Fernø *et al.* (2010).

3.1. Polymer gel for conformance control

Conformance control by polymer gel was studied on core plugs, using all four outcrop core materials. The polymer gel experiments were performed with some variations, and important aspects in the experiments are summarized below.

Experimental design and preparation

Gel system and chemical concentrations

The same polymer gel system was used in all experiments, and was mixed according to **Figure 5** (**Chapter 2**). The solvent was synthetic Ekofisk brine (4% NaCl, 3.4% $CaCl_2$, 0.5% $MgCl_2$ and 0.05% NaN_3). The polymer (Ciba Alcoflood 935) was dissolved in the brine at 5000ppm (0.5%) polymer concentration. The cross-linking agent was Cr(III)-Acetate, received from the manufacturer in powder form. The powder was dissolved in water and added to the polymer solution at 417ppm active Cr(III)-Acetate concentration (0.04%).

The gel was cross-linked at 41^{o}C for 24 hours (five times the gelation time). *In-sit*u cross-linking was performed in some cores, by incubating fractured core plugs in a heating cabinet after injection of immature gel (gelant). Mature gel was more frequently used, where bulk volumes of gel were placed in buffers or beakers and cross-linked at 41^{o}C prior to experiments. The Cr(III)-Acetate-HPAM gel system was extensively described in terms of rheology and chemistry prior to this thesis, see e.g. Liu and Seright (2000), Sydansk *et al.* (2005), Vargas-Vasquez *et al.* (2007), and references within.

The fracture aperture

Some core plugs were fractured longitudinally before use in polymer gel experiments. A band saw was used to create smooth fractures. Open fractures of 1 mm aperture were created by placing POM (polyoxymethylene) spacers between the core halves during assembly. These fractures have a significant effect on fluid flow, and fracture permeability was calculated to be approximately 84 400 Darcy, using the cubic law of Witherspoon *et al.* (1980): **Equation 1** may be used to estimate the permeability of open, smooth-walled fractures, with parallel fracture faces. Fracture permeability is in this case only dependent on the fracture aperture, *b*:

$$k_{frac} = \frac{b^2}{12} \qquad \textbf{Equation 1}$$

Fractured core setups

The experimental setups varied in this work, and different core setups were required for use in the various applications. Some important core setups are summarized in this section. In **Paper 5,** specially designed end pieces were placed on the core inlet end face to separate three injectors: the gel injector (fracture only) and the fluid injectors (matrix only). The setup ensured conformance control by gel in the fracture, without reducing injectivity at the inlet end face, thus fluids could flow into the matrix without external obstructions during chase floods. To separate fracture and matrix production at the outlet, *matrix taps* could be utilized. Matrix taps are fittings or tubing put in direct contact with the rock surface that could be used as additional outlets during fluid flow. They were also used as inlets during immature gel injections, where the goal was to increase the gel saturation in the matrix. Matrix taps were implemented in most cores in **Paper 1**. The majority of cores used in **Paper 1**, **Paper 2**, **Paper 5** and **Paper 6,** and some cores in **Paper 4**, were coated in epoxy prior to experiments. Epoxy coating enabled core flooding at elevated pressures without using a core holder, and was used 1) when the cores were moved between setups and disturbances in overburden pressure could not be risked (**Paper 1**), 2) during spontaneous imbibition, where only a portion of the core was open to flow (**Paper 2**) and 3) during *in-situ* imaging by MRI, where two-phase flow in the core matrix was monitored during injection of gel, foam, surfactant and water (**Paper 4**, **Paper 5** and **Paper 6**).

Gel resistance to washout

Polymer gel resistance to washout during chase floods largely controls the success of the conformance control treatment, and was evaluated experimentally in **Paper 1**. Immature or mature gel was placed in fractured cores, and rupture pressures and subsequent pressure drops across the cores were measured during waterfloods. The experimental steps are summarized in **Figure 8**, and detailed experimental description is found in **Paper 1**.

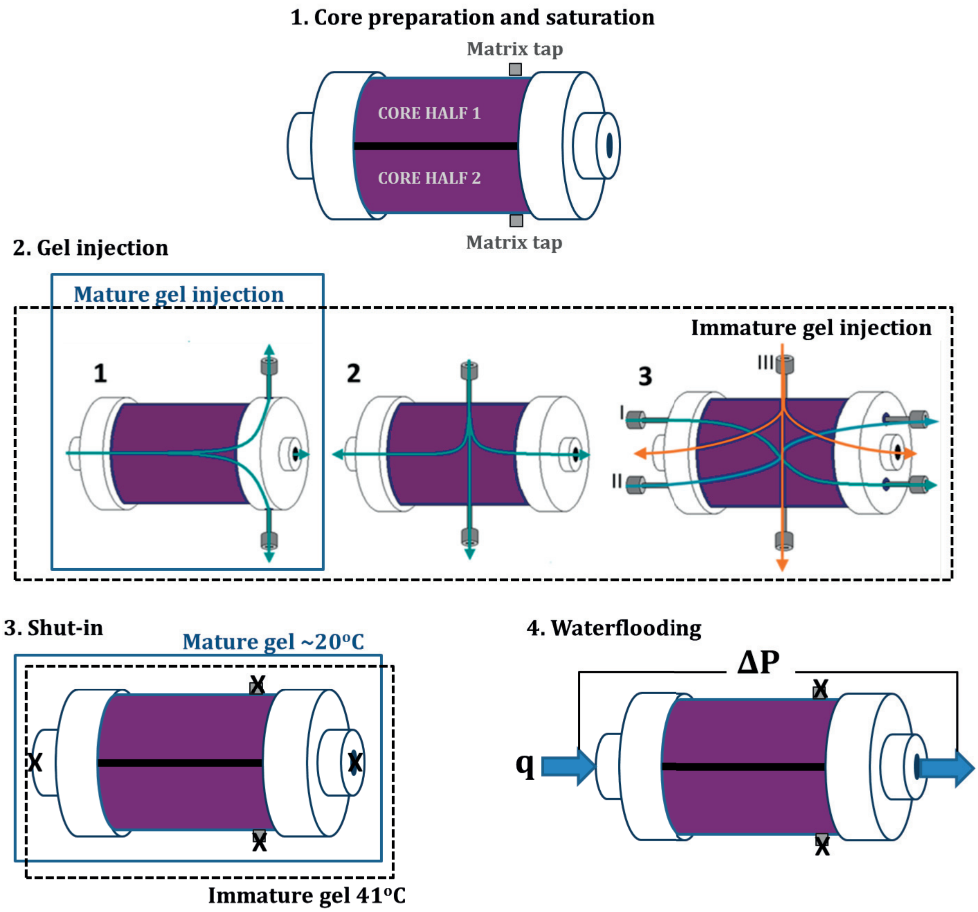

Figure 8: The experimental steps involved in investigation of gel resistance as a function of gel maturity during injection.

The first step was immature and mature gel injection into fractured core plugs to reduce fracture permeability and improve conformance during chase floods. Gelant (immature gel) was injected into the core matrix through the matrix taps, using different injection schemes to control the gelant saturation in the matrix (**Figure 8, middle**). Mature gel flowed in the fracture only during injection, due to its structure. Gel dehydration by water leakoff occurred during propagation through the fracture: water that left the gel progressed through the matrix and was produced through matrix taps and recorded. After gel placement, the cores were shut-in for 24 hours at ambient temperature (mature gel), or an elevated temperature to allow *in-situ* gelation (immature gel). Chase waterfloods were initiated with all matrix outlets closed, hence water flowed through the gel filled fracture. Rupture pressures, and subsequent fracture permeability reduction provided by the gel, was measured.

Comparable rupture pressures were achieved after placement of gel at both maturities. Rupture pressures measured after placement of mature gel in fractures were stable and predictable. Increasing rupture pressures were observed when the injected gel volume increased, with some dependency on gel placement rate. Rupture pressures after immature gel placement and *in-situ* gelation were less predictable, and highly dependent on the gel saturation in the matrix. Ganguly *et al.* (2002) observed that gel did not form when gel was placed in the fracture without penetrating the adjacent matrix. Our findings support this, and gel was in many cases not formed when gelant saturation in the matrix was low. The lack of gelation represents a concern in reservoirs where gelant entry into porous rock is inhibited: 1) at oil-wet conditions, where a threshold pressure must be overcome to invade the matrix, 2) in rock of narrow pore throat radii, for example chalk, and 3) in far-wellbore applications in fractured reservoirs, where gelant channel through fractures at a low pressure gradient.

In some cores of Bentheimer sandstone core material, rupture pressures measured after *in-situ* gelation applications were close to zero, and interactions were observed between the rock and gelant, which caused incomplete cross-linking, i.e. gel did not form. Previous works have described similar effects, especially in sandstone (Stavland and Nilsson 1995, Wilton and Asghari 2007, Seright 2009), and proposed that *in-situ* gelation did not occur due to chromium retention due to precipitation/adsorption. Wilton and Asghari (2007) and Seright (2009) performed experimental steps to force gelation, and the core matrix was preflushed with chromium before gelant injection, or gelant was placed with chromium overload.

Chase waterflooding continued after the gel ruptured. The gel blocking capacity after rupture was dependent on gel maturity during placement, because maturity controls how gel behaves during injection, and consequently how it deposits in the fracture. Gel maturity during placement controls: 1) whether gel extrudes through porous rock, thereby creating a gripping effect between fracture and matrix, and 2) water leakoff during propagation through a fracture, which control the distribution and size of wormholes. Mature and immature gel injection both resulted in improved conformance during waterfloods, however, fracture permeability reduction by mature gel was more efficient. The improved blocking capacity from mature gel placement may be explained by the occurrence of wormholes: when the rupture pressure is reached during chase floods, the fresh gel residing in wormholes is presumably displaced, while the dehydrated gel surrounding the wormholes still resides in the fracture. Thus, the water flowing through the gel-filled fracture is contained in the narrow flow channels constituting the wormholes. The gel forming in the fracture after immature gel injection is of original composition, and is prone to dehydration caused by the induced pressure gradient during chase floods. Gel erosion occurs in a different manner, and may open larger sections of the fracture to flow. **Figure 9** illustrates how the gel maturity during placement influences gel formation in the fracture.

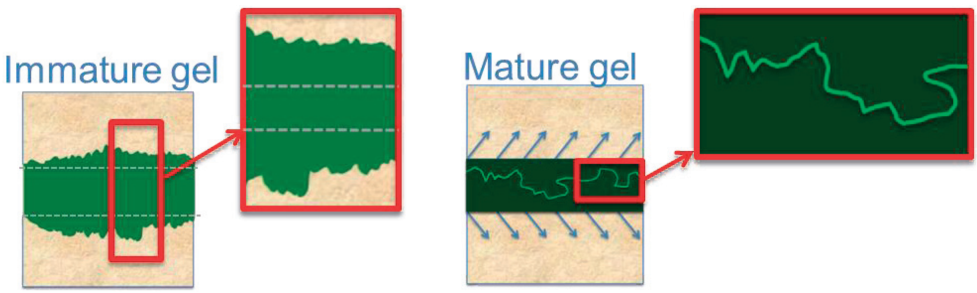

Figure 9: Left: Immature gel is assumed to flow into the adjacent matrix as well as through the fracture during injection. The gel that forms in and around the fracture is of uniform composition. Right: Mature gel cannot flow into porous media, but solvent leakoff during placement may increase the concentration of the gel. Gel of injected composition is present in narrow flow channels (wormholes) through the highly concentrated gel.

Capillary influence on gel stability

Paper 1 use core plugs saturated with water only in experiments, which is often the case when investigating polymer gel behaviour on core scale. Using core plugs with single phase saturation by the gel solvent to simulate the reservoir matrix disregards basic two phase effects. Relative permeability and capillary pressure are of importance during multi-phase flow in fractured reservoirs, and can also affect the success of conformance control. Experiments were performed to evaluate the influence of capillary effects in a strongly water-wet matrix on the properties of an aqueous gel. The work is detailed in **Paper 2.**

Spontaneous imbibition in gel

Spontaneous imbibition experiments, applying all faces open (AFO) boundary conditions were first performed in strongly water-wet rock to evaluate the rate of water extrusion from the gel by capillary spontaneous imbibition. Portland chalk core plugs were directly saturated by mineral oil under vacuum and submerged in mature gel. Imbibition, visible by oil production from the core, commenced shortly after submersion in gel, and continued until the residual oil saturation was reached.

Direct visual recording of produced oil as a function of time was challenging in gel/oil AFO applications. In water/oil systems, AFO spontaneous imbibition experiments are relatively simple to perform, and direct visual recording of produced oil is often possible, e.g. using Amott imbibition cells. When core plugs were submerged in mature gel, a dehydrated gel layer formed on the core surface during imbibition, and produced oil was trapped in the gel layer or the overlying gel for a long period of time. **Figure 10** shows parts of a chalk core submerged in gel during AFO spontaneous imbibition experiments. The oil drops seen above the core surface were not rapidly floating to the surface as they would in water/oil applications, but were captured within the gel for hours, and sometimes for the remainder of the experiment. By visual inspection, the core imbibition process seemed slow and the image of the core and surrounding oil drops remained virtually unchanged for hours, with oil drops on the surface growing and random oil drops detaching from the core.

Figure 10: A chalk core surface during imbibition in mature gel. Oil drops were captured in gel for a long period of time, and the saturation could appear to be static for hours at a time.

Several experimental approaches were tested to measure the oil recovery rate during imbibition, but were not accurate. These include continuous weight recordings of the core and gel, and separation of bulk gel and trapped oil by centrifuging or chemical dissolution. An experimental setup was finally tested, where the core was removed from the gel bath at given time steps and weighed with the dehydrated gel film on its surface. Oil production as a function of time was successfully measured and reproduced through several experiments, applying this method. The experiments were recognized to hold a high uncertainty, because removal of the core from the gel bath disrupted imbibition and disturbed the boundary conditions during the experiment.

A new experimental setup for measuring co-current imbibition was at the same time developed at the University of Bergen by Haugen *et al.* (2014). Their setup was an adaption to two ends open (TEO) boundary conditions, called *TEO free spontaneous imbibition* (TEOFSI), where one core end face was in contact with water (the imbibing fluid) and the production end face was in contact with oil. The capillary back pressure was not acting on the end face contacted by oil, hence oil in place could be produced from the core without resistance. In water/oil imbibition experiments applying TEOFSI boundary conditions, a small volume of oil was produced counter-currently at the start of imbibition, but all subsequent production of oil occurred co-currently. This was also observed when the setup was tested in gel/oil applications, and direct visual recordings of co-current oil production were possible for the majority of spontaneous imbibition. The measured imbibition rate was slower when cores were submerged in gel, than in water/oil systems, although the flow properties within the core remained unchanged. This was caused by a concentrated layer of gel forming on the imbibing core surfaces during gel dehydration, and acting as a low permeability barrier to flow. A schematic of the experimental setup is shown in **Figure 11**.

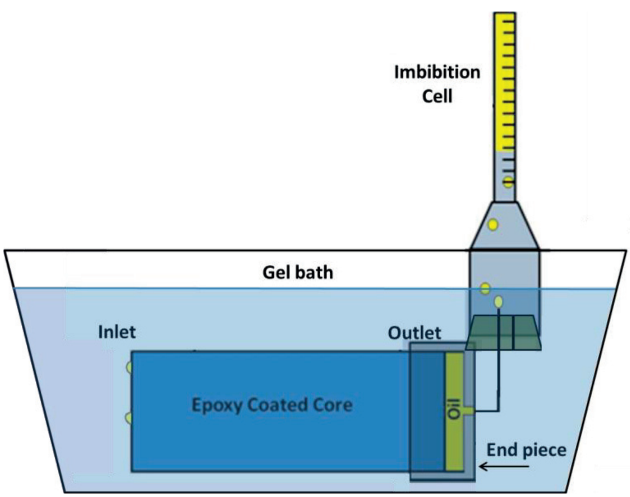

Figure 11: Experimental setup for TEOFSI spontaneous imbibition experiments in gel. The figure was modified from Haugen *et al.* (2014).

Gel shrinkage

Gel dehydration and shrinkage, caused by spontaneous imbibition, were the most important implications of this experimental work. The polymer gel network, in which the strongly water-wet cores were submerged, was inflated by ~99.5% water, and when capillary forces in the matrix attracted gel-bound water, a significant decrease in gel volume followed.

Up to 99% of the initial gel volume was lost due to spontaneous imbibition of water from gel in our experiments, with severe implications for gel blockage capacity during chase floods. Polymer gel is more efficient in reducing fracture permeability if a significant portion of the fracture volume is gel-filled. In a completely gel-filled fracture, spontaneous imbibition reduces the gel volume over time and inhibits gel from blocking the entire fracture cross-section. Parts of the fracture volume can conduct fluid flow, and the gel treatment becomes less efficient. **Figure 12** illustrates the effect of gel shrinkage on fluid flow in a fractured system. The increase in fracture permeability caused by gel shrinkage was calculated using Darcy flow calculations for parallel layers, detailed in **Paper 2** and was highly dependent on the fracture aperture for gel shrinkage below 20% of the initial gel volume. 99% shrinkage from spontaneous imbibition was detrimental to gel conformance control treatments in all fracture apertures, and fracture permeabilities returned to the original levels. The demonstration of capillary influences on gel behaviour has important implications for gel use in fields where the wettability tends towards water-wet, and may help explain why some gel treatments are less successful. Several authors have reported that gel treatments have in some cases been less effective than expected in reducing producing well water cut, or that gel treatments have lost efficiency with time (White *et al.* 1973, Seright *et al.* 2003, Portwood 2005).

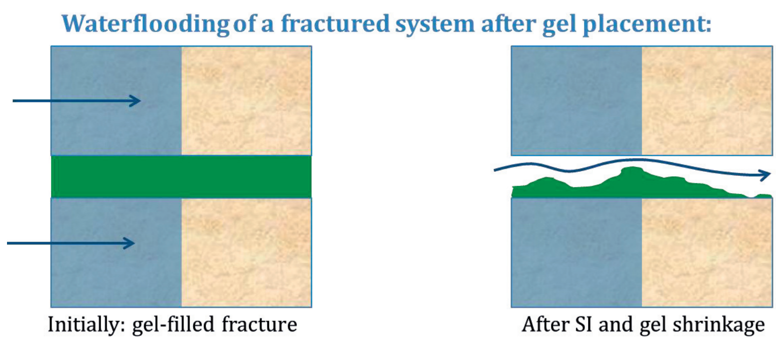

Figure 12: left: when low permeable polymer gel is placed in a fracture to reduce fracture permeability, water may enter into matrix blocks at an increased viscous pressure gradient during chase floods. Right: gel shrinkage cause parts of the initially gel-filled fracture to re-open to flow, and gel blocking efficiency is reduced. Waterflood commence through the open portion of the fracture.

Gel placement and flow diversion at oil-wet conditions

The experiments in **Paper 2** verified that two-phase flow functions in a matrix may influence the properties of an adjacent gel. At oil-wet conditions, capillary forces limit the intrusion of water to the matrix, and spontaneous imbibition of water from aqueous gels (and thereby loss of gel integrity) is less likely. Oil recovery by waterflooding is not efficient in fractured, oil-wet formations for the same reason, and they are attractive targets for EOR operations. Experimental results from **Paper 5** are presented, where conformance control by polymer gel was investigated as the first step in an integrated EOR method to improve waterflood efficiency in an oil-wet limestone core ($I_{AH} = -0.7$). All experimental steps were monitored through *in-situ* imaging by a medical CT scanner.

Waterflooding

A water preflush was first performed, where water was injected directly into the matrix through two separate injectors. Total oil recovery was 4%OOIP, and mainly attributed to the injector positions. At dimensionless length $X_D = 0.1$, visualized by the CT, water broke through to the fracture and channeled to the outlet end. No further oil recovery from the matrix was seen during water injection prior to conformance control.

Gel placement

Mature gel was injected into the fracture to reduce fracture permeability and increase waterflood efficiency during chase floods. During injection, the gel reached the outlet after one fracture volume (1FV) injected, suggesting that water leakoff did not occur. This was verified by the CT scans: no saturation changes in the matrix were detected. This may be explained by capillary forces that were present in the matrix, and prevented the entry of gel-bound water below a capillary threshold pressure. The gel did therefore not dehydrate during extrusion through the fracture, unlike **Paper 1**, where capillary forces were not present and gel dehydration was controlled by the pressure gradient across the fracture.

Chase waterflooding

Chase waterflooding was performed after gel was placed in the fracture for conformance control. Gel in the fracture was of the original composition and had not concentrated or allowed wormhole formation during extrusion. The recovery during chase water injection was 7%OOIP and 15%OOIP for the core halves, respectively, despite gel in the fracture being of original composition. Injected water entered the fracture at $X_D = 0.4$, after which no further oil recovery was seen. **Figure 13** shows CT images taken after low rate polymer gel injection (**Figure 13**, top), and chase waterflooding (**Figure 13**, bottom). A significant change in saturation was seen at the inlet end of the core plug subsequent to chase waterflooding, especially in the upper core half. The displacement front, visualized by CT, was quickly dispersed during water injection. Hence, oil was mainly displaced from the matrix close to the fracture.

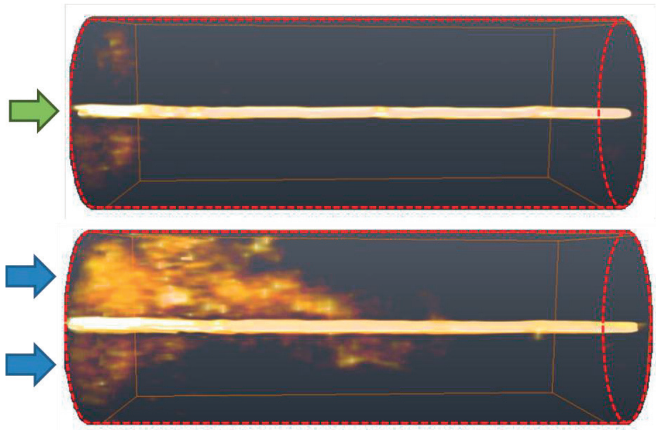

Figure 13: CT images of an oil-wet limestone core after low rate polymer gel placement (top), and chase water injection (bottom).

Measuring the rate of solvent leakoff from the gel is important. The leakoff rate describes the concentration of the gel in the fracture, and thus its ability to divert chase fluids. It also gives important information on gel propagation in a fracture, and the distance of gel intrusion in a formation can be assessed. The general opinion has been that leakoff occur when gel propagate through a fracture at any given rate, and that the limiting factor in achieving leakoff is the pressure needed to enter the fracture. This pressure is largely controlled by the fracture aperture, and does not vary significantly with injection rate. This theory has been verified in several experiments, where core plugs saturated by one phase only have been used. Leakoff was attained at all injection rates, ranging from 6 to 200 ml/h in **Paper 1**, and Seright (2003a) measured equal leakoff rates in cores of 1.5 mD and 650 mD permeability when gel was injected at a high flow rate, and proposed that leakoff of solvent from mature gel was largely independent of changes in permeability and lithology. **Paper 2** and the experiment described above, from **Paper 5**, show that wettability, and wettability dependent saturation functions in the matrix, affect gel behaviour during placement. Thus, leakoff is not solely controlled by fracture properties; matrix properties must also be assessed when considering gel placement in a fracture where the adjacent matrix is saturated by more than one phase.

This thesis clearly shows that the fluid saturation in the matrix, and associated saturation functions, heavily influence conformance control by gel and are important when assessing gel behaviour, during and after placement, in hydrocarbon reservoirs. The transport of gel-bound water into an adjacent matrix depend on wettability, and: 1) may occur by spontaneous capillary imbibition of water from the gel in water-wet systems, without imposing an external pressure gradient on the system, and 2) may not occur in oil-wet systems due to capillary forces in the matrix that do not allow solvent entry during gel injection. This indicates that gel behaviour is dependent on matrix properties, above all wettability. Other matrix parameters, such as pore size distribution (rock type) and saturation also influence the ease at which the water leaves the gel or fracture and goes on to flood the matrix.

3.2. CO_2 flooding

Experiments and preparation

Whole and fractured core plugs were used to study enhanced oil recovery by CO_2 flooding. The core plugs were saturated, drained, in some cases aged and frequently fractured before they were subjected to secondary and tertiary CO_2 floods. CO_2 flooding was performed using CO_2 in gas, liquid and supercritical phase, alone and mixed with surfactant solution to form foam. Temperature and pressure varied between the experiments, from ambient to reservoir conditions; hence, CO_2 was immiscible or miscible dependent on the experimental conditions.

Experiments that involve the use of gas require caution at high pressures and temperatures, due to significant changes in density between ambient and experimental conditions (see **Figure 6**). **Figure 14** shows a generalized experimental setup for CO_2 and N_2-/CO_2-foam injections. CO_2 was in most cases introduced to the system through an accumulator or a piston pump: injection pumps with small volume cylinders were not able to pressurize the highly compressible gas and deliver it to the system without significant uncertainty in the flow rate. The accumulator was placed in a heating cabinet or at room temperature and was filled with CO_2 and pressurized to the experimental pore pressure before injection. It was important that CO_2 reached the specified temperature conditions prior to entering the core, due to significant changes in injected CO_2 density and viscosity as functions of temperature. This was achieved by injecting the gas through a coiled injection tubing of approximately 3 meters length placed ahead of the core in the heating cabinet. In foam applications, N_2 or CO_2-foam was pre-generated through a sandpack or Bentheimer sandstone core (either termed a *foam generator* in the experimental setup) at a gas fraction of $f_g=0.9$ prior to injection. The pore pressure was controlled using a back pressure regulator (BPR) pressurized by a Nitrogen (N_2) gas tank.

Miscible CO_2 injections for EOR in fractured carbonates

Paper 3 investigates the effect of oil composition during CO_2 EOR. Both secondary and tertiary CO_2 injections were performed using n-Decane and a North Sea chalk reservoir crude oil as the oil phase. Whole and fractured chalk core plugs at different wetting conditions were used. The experiments were performed at elevated temperature and pressure conditions of 40^oC/95bar (n-Decane) and 75^oC/208bar (crude oil).

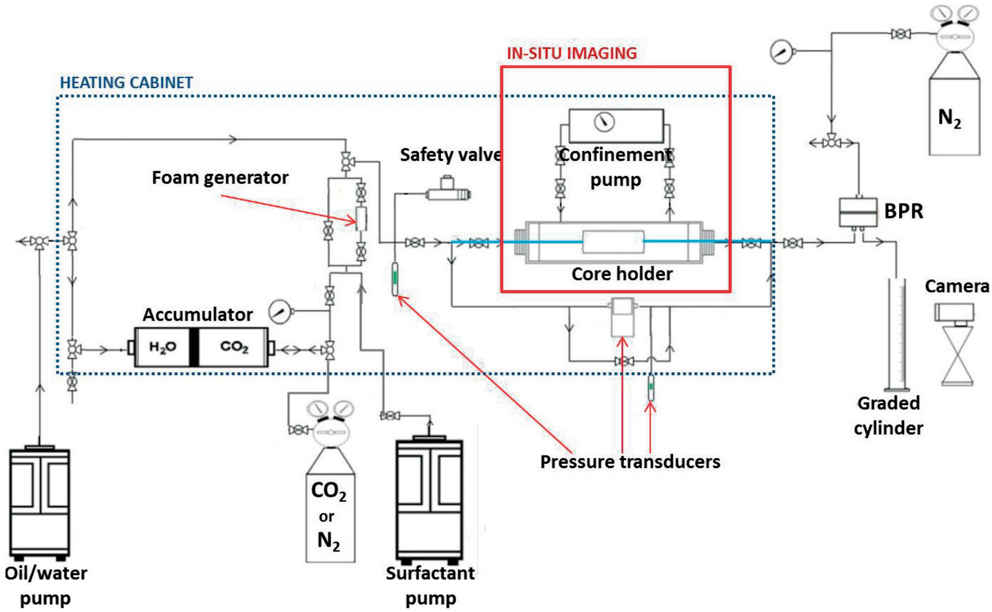

Figure 14: Experimental setup for pure CO_2 and N_2-/CO_2-foam injections.

Influence of oil composition on secondary CO_2 flooding

Secondary CO_2 floods yielded high oil recoveries, where the end point depended on the oil composition in the core. When the oil phase was a one-component mineral oil (n-Decane, $C_{10}H_{22}$), oil recovery of 96%OOIP was reached during CO_2 flooding of both fractured and whole cores, at 4PV CO_2 injected in the fractured system, compared to 2PV (non-fractured). The high recovery in the fractured cores were mainly products of relatively large fracture volumes compared to the total core pore volumes, which made contributions from diffusion a major recovery mechanism. Oil recovery decreased and prolonged tail production was observed when complex hydrocarbons (North Sea crude oil) constituted the oil phase, due to the development of multi-contact miscibility. Diffusion was less efficient as a recovery mechanism in crude oil systems that contained both light and heavy hydrocarbon components: light hydrocarbons were efficiently recovered, by vaporizing into the CO_2. Heavier hydrocarbon components were not as easily dissolved into the gas phase, and were produced during tail production or remained in the core to constitute the residual oil saturation. Visual inspections of the effluents indicated a variance in produced oil composition, and the crude oil produced early in the experiment (after CO_2 breakthrough) was less viscous than the oil produced after significant tail production. Although dependent on CO_2 density, oil components up to C_{30} are considered to be relatively efficiently extracted by the gas phase (Silva and Orr 1987). The North Sea crude oil consisted of 28% C_{30}+ components, which explains the higher residual oil saturation endpoint.

Tertiary CO_2 flooding at different wetting conditions

CO_2 floods were implemented in tertiary recovery mode, after waterflooding, in **Paper 3** and **Paper 4**. A significant difference in recovery was observed compared to secondary CO_2 floods. Viscous recovery of oil was attained by CO_2 flooding in whole cores, while fractured cores were severely affected by the presence of water films: water prevented contact and mixing between the CO_2 and oil phase, hence recovery by diffusion was low. **Figure 15** shows the development in oil saturation during waterflooding and tertiary CO_2 flooding of fractured core plugs at strongly water-wet and neutral-wet conditions (**Paper 3**), and at oil-wet conditions (**Paper 4**).

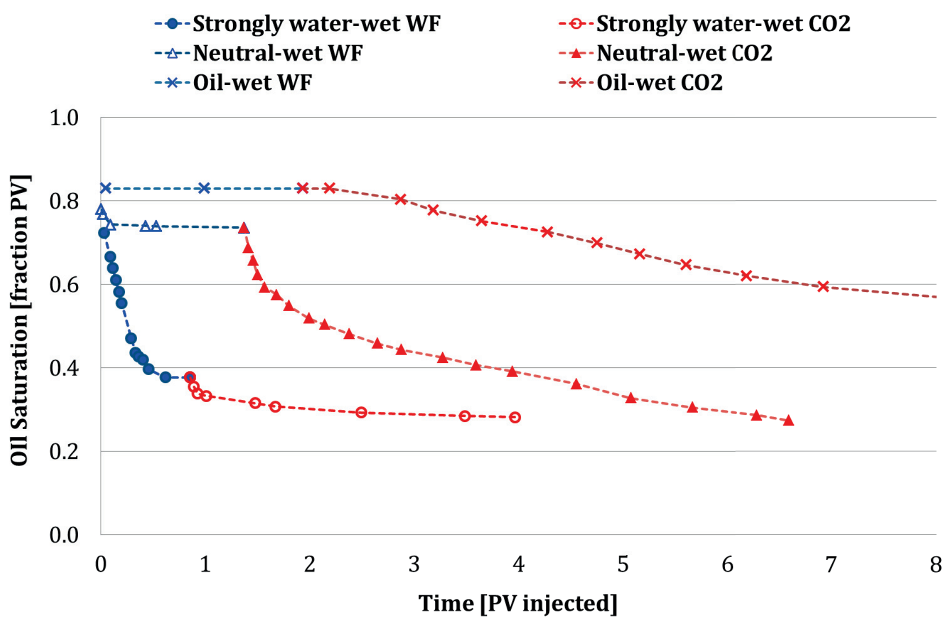

Figure 15: Decrease in oil saturation during waterflooding (blue) and miscible CO_2 flooding (red) in fractured, strongly water-wet (circles), neutral-wet (triangles), and oil-wet (crosses) cores. The water-wet and neutral-wet core data are from Paper 3, and the oil-wet data set from Paper 4.

Wettability controls the distribution of fluids in the pore space and influenced oil recovery during tertiary CO_2 floods in fractured cores: efficient waterflooding of the strongly water-wet core resulted in pronounced water shielding and an inefficient subsequent CO_2 flood. At neutral wetting conditions, waterflooding of the fractured core produced less oil. The tertiary CO_2 flood could therefore contact, and mix with, the oil phase in several locations, and produced 59%OOIP of additional oil. The total oil recovery from the strongly water-wet and neutral-wet cores was comparable. In the oil-wet core, waterflooding and CO_2 flooding were both inefficient. This experiment is not directly comparable to the other two, due to a closed fracture, n-Decane in place of crude oil, and lower experimental pressure conditions (lower CO_2 density and viscosity), but illustrates an important point: several pore volumes of CO_2 were required to reach the residual oil saturation during miscible gas floods of fractured core plugs in both **Paper 3** and **Paper 4**. **Figure 15** shows that residual oil saturations were not reached during injection of close to 6PV CO_2 for the neutral-wet and oil-wet cores, because:

- ❖ The high mobility of the gas phase caused fracture channeling and a swift breakthrough of gas.
- ❖ Molecular diffusion was the main recovery mechanism in fractured core plugs, and is a slow process.

Miscible and immiscible CO_2-foam for tertiary oil recovery

Mixing CO_2 with a surfactant solution to form foam decrease the mobility of the flowing gas phase and improve conformance by contributing a viscous component to the core flood. Foam constituents (gas and surfactant) can be diverted into the matrix to efficiently recover oil. CO_2-foam flooding was investigated for tertiary EOR in fractured, strongly water-wet and weakly oil-wet carbonates in **Paper 4**. Waterflooding at oil-wet conditions was inefficient (0-24%OOIP) and the potential for enhanced oil recovery during tertiary recovery operations was high.

The oil-wet core plugs had closed, longitudinal fractures, and the fracture permeability ranged from 0.3 to 2Darcy. CO_2-foam floods were performed at both miscible and immiscible conditions after waterflooding to improve conformance and enhance oil recovery. **Figure 16** shows the oil recovery achieved during tertiary injection of miscible (red lines) and immiscible (blue lines) CO_2-foam. Oil recovery from CO_2 flooding without conformance control is shown for comparison (black line). Miscible foam floods recovered more oil, at a higher rate, compared to the other investigated applications, and the recovery end point was reached within 2PV foam injected. Up to 35PV of CO_2-foam were injected to reach the endpoint in immiscible applications. *In-situ* imaging by CT revealed that gas did not enter the matrix to recover oil during immiscible N_2-foam injections at oil-wet conditions. Oil recovery during immiscible foam injection was hence attributed to the aqueous surfactant solution, which entered the core to displace oil at an increased viscous pressure gradient provided by the foam. This results support the findings of Haugen *et al.* (2012), who reached the oil saturation end point after injection of more than 40PV foam at oil-wet conditions, suggesting that the oil was recovered by surfactant solution only.

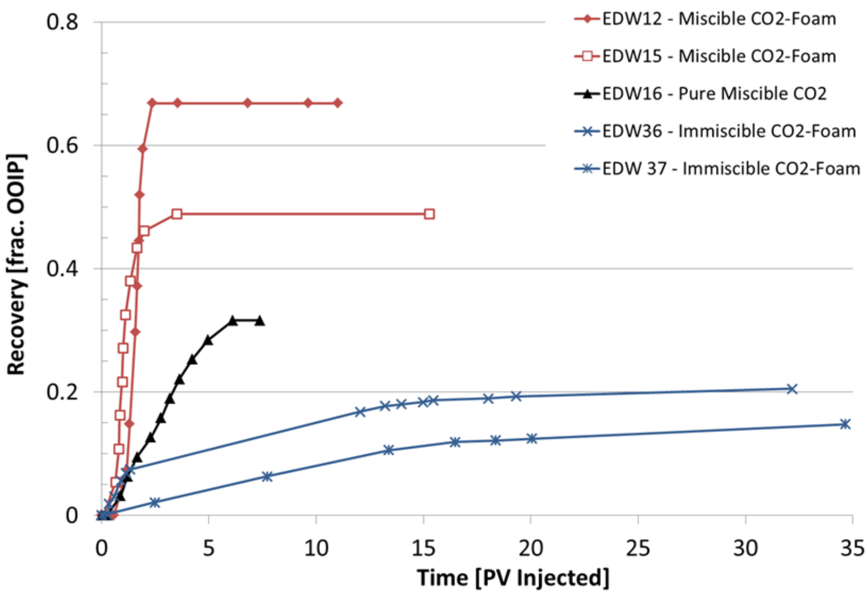

Figure 16: Efficiency of tertiary miscible and immiscible CO_2-foam floods in fractured, oil-wet limestone cores. The black line illustrates tertiary recovery by pure, miscible CO_2.

Foam applications in fractured systems were more efficient when both foam constituents entered the matrix, i.e. in miscible foam applications. Still, more than 2PV foam was needed to reach maximum recovery: displacement of matrix oil required strong foam to be generated in the fractures first, and provide the viscous pressure needed to enter the matrix. Conformance control through fracture permeability reduction prior to foam injection may enable entry of foam to the matrix at injection onset, thus fewer PV are needed to recover oil. Integrated EOR methods that combine near-wellbore and in-depth conformance technologies with chemical EOR technologies, are of growing interest in the oil industry (Manrique *et al.* 2010).

3.3. Integrated EOR

Paper 5 experimentally investigates an integrated EOR approach, where the first step is polymer gel injection to reduce fracture permeability. Conformance control by polymer gel enables fluids to enter matrix blocks at increased pressure gradients during chase floods, thereby enhancing oil recovery.

The experiments were performed using fractured, oil-wet limestone core plugs. Capillary forces at the oil-wet conditions prevented water entry to the matrix: spontaneous imbibition of the oil phase was observed during wettability measurements, using the Amott method, while the Amott water index was zero. No imbibition of water suggested that aqueous gel could be used without expecting gel shrinkage after placement. *In-situ* imaging by a medical CT scanner, as well as magnetic resonance imaging in a low-field 8 MHz MRI (DRX) machine, enabled a detailed view of the fluid distributions during all experimental steps.

Waterflooding

Waterflooding of the fractured system was performed before implementation of EOR methods, to establish baseline recovery. Oil recovery of up to 10.7 %OOIP was observed, primarily near the inlet end. Waterflood oil recovery was caused by forced injection of water into the matrix through specially designed end pieces (ref. **Chapter 3.1** and **Paper 5**). Water quickly channeled through the matrix and reached the open fracture. No further oil recovery from the core matrix was detected after water breakthrough to the fracture.

Gel placement

When polymer gel propagates through a fracture, solvent leakoff may occur. During low rate gel injection in one of the oil-wet cores (I_{AH} = -0.7), leakoff was not observed, due to a capillary threshold pressure in the matrix. Four additional gel placements were performed, where the pressure differential between the fracture and matrix was high enough to enable water entry to the core matrix, and oil was consequently displaced. Up to 34%OOIP was recovered from the matrix during polymer gel injection, caused by two factors: 1) the fracture volume in the experiment was large relative to the core pore volume, thus the injection of less than ten fracture volumes of gel corresponded to one pore volume injected. 2) The injection pressure, which was the driving force for water leakoff, was high. During *in-situ* monitoring by MRI, the core had to be mounted vertically. Polymer gel was injected upwards through the fracture, and induced a high differential pressure. Polymer gel injection into fracture reservoirs is not expected to contribute to significant oil recovery, because 1) the water leakoff rate, and hence the volumetric oil displacement rate during gel placement, is low, 2) the fracture volume constitute a smaller part of the reservoir pore volume, and 3) injection time is limited to days or weeks.

Chase floods

Water, foam and surfactant chase floods were performed in the fractured core plugs after implementation of conformance control by polymer gel. The *in-situ* displacement processes were visualized by CT and MRI. Dispersed displacement fronts were observed during chase floods by water and surfactant solution, and oil was mainly displaced from the matrix close to the fracture. Chase flooding by immiscible CO_2-foam was performed to mitigate fluid dispersion around the displacement front. The results are described below, and compared to results from **Paper 4**.

Immiscible foam floods for tertiary EOR were investigated in **Paper 4** and **Paper 5**, at analogues core properties and wettability conditions (weakly oil-wet). **Table 3** shows core properties and experimental data for the core plugs. In **Paper 5**, polymer gel was placed in the fracture for conformance control prior to CO_2-foam flooding. In **Paper 4**, foam floods were performed directly after waterflooding: *in-situ* imaging by CT showed that oil was displaced by surfactant solution only at the given wetting conditions. The surfactant was able to flood the matrix at an increased viscous pressure gradient provided by the foam. **Figure 17** shows the *in-situ* development in oil saturation during immiscible CO_2-foam flooding of core EDW36 (no conformance control prior to foam). Oil was mainly displaced from the matrix area close to the fracture, presumably by surfactant solution.

Table 3: Overview of selected core properties, experimental data and results from immiscible CO_2-foam floods in fractured, oil-wet core plugs. CO_2-foam floods were initiated directly after waterflooding in core EDW36 and EDW37, while polymer gel was placed in the fracture for conformance control before foam flooding of core B.

Ref.	Core	Wetta-bility [I_{AH}]	k_{frac} [D]	[1]WF oil recovery	Conformance control prior to foam	Foam inj. [PV]	Recovery [%OOIP]
Paper 4	EDW36	-0.2	1.188	9.3	No	32	20.5
Paper 4	EDW37	-0.2	2.020	23.8	No	30	14.8
Paper 5	B	-0.2	[2]84400	10.7	Polymer gel	12.7	28.0

[1]WF = waterflood.

[2]fracture permeability calculated from Witherspoon et al. (1980), Equation 1.

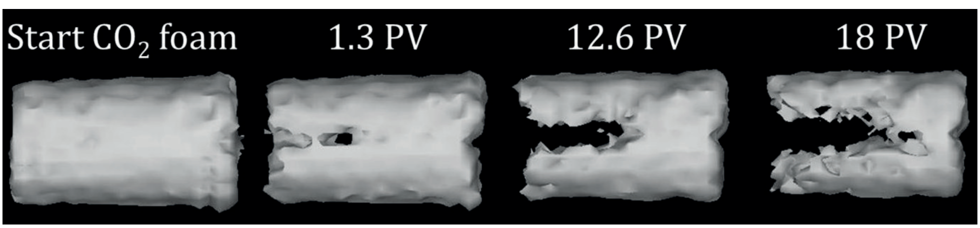

Figure 17: Development in oil saturation during immiscible foam flooding of fractured, oil-wet core plug EDW36. Foam was injected left to right.

In core B, polymer gel was placed in the fracture prior to CO_2-foam flooding, and both foam constituents (gas and surfactant solution) entered the matrix during the foam flood. A higher injection pressure was observed during CO_2-foam flooding of core B than for cores EDW36 and EDW37, which indicated that gas and surfactant both propagated through the porous matrix to displace oil. Immiscible foam injection after conformance control by polymer gel provided 28%OOIP additional oil recovery during 12.7 PV foam injected. In comparison, recovery factors of 14 to 20%OOIP were obtained during injection of more than 30PV foam in EDW36 and EDW37. The initial fracture permeability for core B was high, at approximately $8.4*10^4$Darcy, calculated by **Eq.1**. Core plugs EDW36 and EDW37 had closed fractures with lower fracture permeabilities of 1 to 2Darcy, as well as higher oil saturations at the onset of CO_2-foam injection, and were as such more likely to obtain high oil recovery factors by foam flooding. Improved sweep efficiency was visually observed by MRI during foam injection (**Paper 5**): foam flooding was observed to give more stable displacement fronts compared to e.g. chase waterflooding (**Figure 13**) in oil-wet media, and was less prone to viscous fingering. The combination of two established EOR methods to improve conformance in both the fracture and matrix has important implications for future work on developing efficient integrated EOR schemes.

3.4. Measurement of two-phase flow functions during waterfloods

When two, or several, immiscible phases flow simultaneously in a porous medium, saturation functions are of importance. Relative permeability and capillary pressure have several times in this thesis been shown to influence fluid flow and EOR processes in porous media. Relative permeability is "a direct measure of the ability of the porous system to conduct one fluid when one or more fluids are present. These flow properties are the composite effect of pore geometry, wettability, fluid distribution and saturation history" (Anderson 1987c). The notion of capillary pressure in a porous medium is based on the curvature of the interface between two immiscible fluids: whenever the interface is curved, the pressure will abruptly increase across it to balance interfacial tension (IFT). This pressure jump is the capillary pressure. "The radii of curvature of the interface, and hence the capillary pressure, are determined by local pore geometry, wettability, saturation and saturation history" (Anderson 1987a). Relative permeability and capillary pressure are heavily influenced by the method of measurement, and different curves may be obtained from static (steady state) methods compared to dynamic (unsteady state) methods. Accurate methods for measuring relative permeability and capillary pressure during dynamic floods are always in demand. An experimental method for determining imbibition relative permeabilities from a single waterflood is presented in **Paper 6**.

Steady state methods are sometimes preferred for relative permeability measurements: reservoir simulators require input relative permeability curves that are applicable over a large saturation interval, which steady state methods provide. The methods require simultaneous injection of two immiscible fluids, and equilibrium between inflow and outflow fluid ratios at several saturations. The challenges in using steady state methods follow from this: 1) the method is time consuming, and 2) simultaneous injection of two immiscible fluids mixes the drainage and imbibition processes, and may thus fail to capture the true properties of a pure imbibition (or drainage) process (Eleri *et al.* 1995). Unsteady state methods are based one fluid displacing another (immiscible fluid) using a constant injection rate or pressure. Continuous measurements of pressure and saturation during oil- or waterflooding are used to generate relative permeability curves. Saturation from effluents and absolute differential pressure over a core sample are commonly used as basis for calculation, applying the JBN method (Johnson *et al.* 1959) or similar (Jones and Roszelle 1978) to generate the relative permeabilities. The approximations implied in the calculations are coarse and do not account for capillary pressure, capillary end effects and the rapid pressure changes across the displacement front. Average core saturation measured from fluid effluents collected at the core outlet provide relative permeability curves over a limited saturation range, because calculation methods are only valid during two-phase production, in addition to adding significant uncertainty to the calculations. Many recent unsteady state methods (Jennings Jr. *et al.* 1988, Chardaire-Riviere 1992, Mejia *et al.* 1995, Goodfield *et al.* 2001, Schembre and Kovscek 2002, Lackner and Torsaeter 2005) utilize *in-situ* measurements of either saturation or pressure, and use numerical methods to estimate the other. Relative permeability curves are then generated by simulation.

A technique was developed by Kolltveit *et al.* (1990), which enabled simultaneous *in-situ* measurement of both pressure and saturation. They used Nuclear Tracer Imaging (NTI) to quantify core saturation during flow and measured absolute fluid pressure at two different locations along the core length. Simultaneous measurements of saturation and pressure *in-situ* were used to calculate relative permeability. In **Paper 6** we developed this method further: we used high resolution Magnetic Resonance Imaging (MRI) to monitor saturation, and local phase pressures were measured in two locations by implementing two oil-wet and two water-wet semi-permeable discs in the core. *In-situ* monitoring of saturation enabled accurate descriptions of fluid distribution in different locations. Continuous measurement of oil/water phase pressures during waterfloods, in conjunction with saturation data, produced dynamic capillary pressure curves without approximations. Relative permeability was calculated explicitly using measured capillary pressure, saturation and rate data.

Chalk cores at different wettability conditions were subjected to this method, using the experimental setup sketched in **Figure 18**. **Figure 19** shows the extracted water and oil relative permeability curves for a strongly water-wet Portland chalk core, compared to conventional relative permeabilities from steady state experiments.

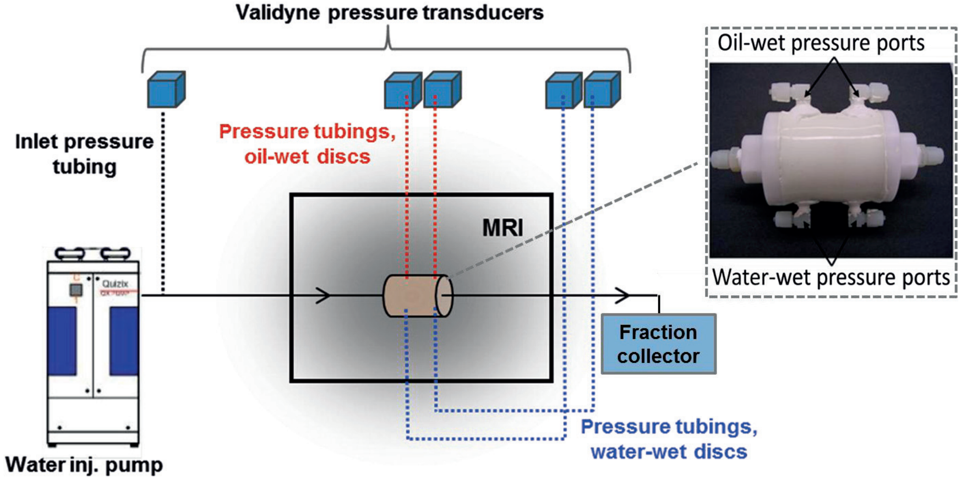

Figure 18: Experimental setup for monitoring *in-situ* saturation development and measuring phase pressures during waterfloods. The collected data set can be used to extract unsteady-state relative permeability curves, or to estimate core wettability during core floods, because the phase pressure response separates the spontaneous and forced part of a water imbibition process (Brautaset *et al.* 2010).

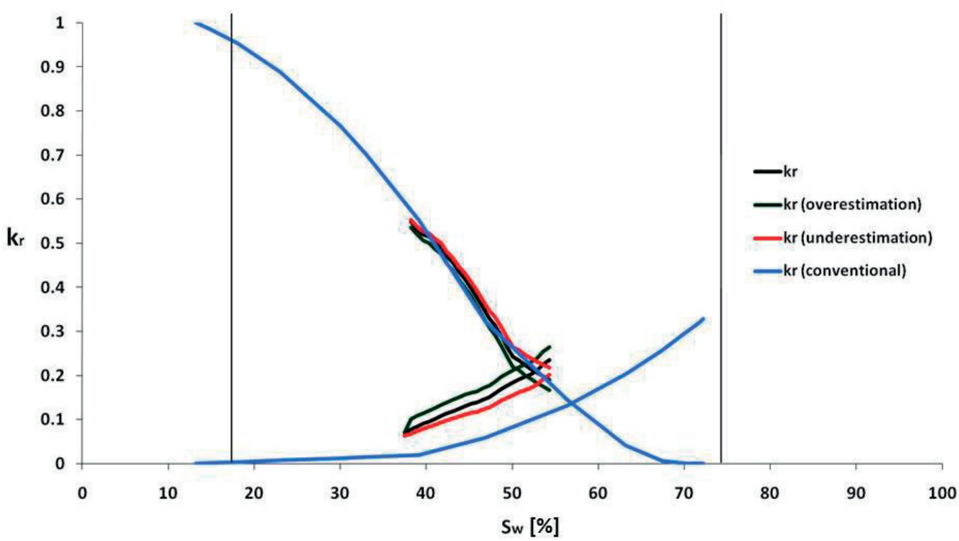

Figure 19: Relative permeabilities measured during waterflooding of a strongly water-wet chalk core. The differences in water relative permeability curves between the unsteady state and conventional data is believed to be caused by spontaneous capillary imbibition of water into the core at water-wet conditions. Spontaneous behaviour is suppressed by the characteristics of a steady state method, while enabling higher water flow rates at unsteady states.

4. Conclusions

This thesis investigated polymer gel and CO_2-foam behaviour in conjunction with oil saturated porous media at different wettability conditions, and improved the understanding of conformance control for EOR in fractured media. The main contributions of the work in this thesis are listed below.

- **The rupture pressures measured after placement of polymer gel in fractures was comparable after placement of immature gel, cross-linked *in-situ*, and mature gel.** Gel blocking ability before rupture in fractured, water saturated core plugs was independent on gel maturity during placement (provided gel formed), hence, injection scheme may be chosen based on other criteria, e.g. desired pressure gradients, injection volume, near-well or far wellbore application, etc.

- **Research on polymer gel behaviour was extended to water and oil saturated media.** Two important observations were made:
  1) Water left mature gel due to capillary spontaneous imbibition into a strongly water-wet adjacent matrix. Spontaneous imbibition was measured applying different boundary conditions, and shown to be slower than for oil/water systems. An important implication of capillary extraction of solvent from gel was gel shrinkage. Up to 99% of the original gel volume was lost during shrinkage, and may help explain why gel treatments in some field applications have lost efficiency with time.
  2) The leakoff effect, where water leaves mature gel during extrusion through a fracture, was not always observed at oil-wet conditions. Capillary forces are present when the matrix is saturated by more than one phase, and a threshold pressure must be overcome for water to enter the matrix at oil-wet conditions. This threshold pressure was in some cases not overcome during gel injection, and the gel propagated through the fracture without dehydrating. This may affect the pressure resistance of the gel, because the gel does not concentrate and form wormholes during extrusion. However, lack of leakoff may also increase the distance a gel is able to propagate to within the reservoir, and increase the in-depth performance of the gel treatment.

- **Miscible CO_2 floods were investigated in fractured systems of varying oil composition and wettability.** A high and fast recovery of oil was achieved when pure mineral oil constituted the oil phase, both in whole and fractured core plugs. When complex crude oil was used, decelerated recovery factors and prolonged tail production was observed during CO_2 floods. Tertiary recovery processes could be less efficient than expected due to water shielding. Water shielding was partly governed by wettability, because wettability controls the post-waterflood saturation and distribution of fluids.

- **Miscible and immiscible CO_2-foam floods were performed to improve conformance and enhance oil recovery during gas floods.** Foam provided an additional viscous pressure gradient that contributed to a more efficient oil recovery from fractured core plugs. Miscible foam injection was the most efficient of all investigated methods in enhancing oil recovery. The injection pressures during miscible foam injection were low without jeopardizing foam quality, which is advantageous for in-depth foam applications on field scale.

- **Proposed recovery mechanisms during immiscible foam flooding were investigated at water-wet and oil-wet conditions.** *In-situ* imaging at strongly water-wet and weakly oil-wet conditions provided important information about immiscible foam flooding. At water-wet conditions, gas entered the matrix and contributed to displacement of fluids. At oil-wet conditions, gas did not enter the matrix, and oil was displaced by the aqueous surfactant solution, which entered the matrix due to an additional viscous pressure gradient provided by the foam.

- **An integrated EOR method was investigated, where placement of polymer gel for conformance control in fractures was the first step.** Polymer gel successfully reduced fracture permeability in oil-wet cores and chase floods were performed at elevated pressure gradients. CO_2-foam was most efficient during chase floods and a stable displacement front was visualized utilizing *in-situ* imaging by CT.

5. Future Perspectives

Polymer gels

Capillary spontaneous imbibition of water from gel into strongly water-wet systems was observed and measured in this thesis. Experiments are being planned to assess the effects of capillary forces on gel at less water-wet or oil-wet conditions, and using immature gel in addition to mature gel. Experiments will also be performed to investigate the influence of saturation functions on the gel placement process. Efforts are currently made to model the exchange of fluids between gel and core surface during spontaneous imbibition, based on the experiments presented in this thesis, and may be extended to include changes in wettability and gel maturity.

Gel applications for use on the NCS must meet certain requirements. First, chemicals (such as chromium) are strongly regulated, which calls for the use of environmentally friendly ("green") gels. Efforts should be made to seek cross-linking agents, in particular, that are compatible with established polymers and applicable according to the PLONOR regulations. Secondly, the polymer gel technology we investigated may be less resistant in reservoirs at high salinity and high temperature conditions. Experiments should be performed to evaluate the efficiency of the given polymer gel system in specific reservoirs.

Previous works have established effects which should be considered when discussing gel treatments in fractures: the establishment of wetting phase bridges (**Chapter 1.1.**) provide transportation of fluids and viscous pressure gradients across transverse fractures, and improve oil recovery at less water-wet conditions. The effect of gel placed in transverse fractures on liquid transport across the fracture (e.g. by wetting phase bridges) should be established.

The experimental setup used for *in-situ* measurement of unsteady state relative permeability (**Figure 18**) offer unique possibilities in conjunction with fractured rock EOR: measuring fluid phase pressures during gel extrusion and chase floods in fractured core plugs may provide useful information on the influence of two-phase functions on the different experimental steps, and for different wetting conditions.

Foams

Experiments using foam for conformance control are presented in **Paper 4**, and are encouraging in terms of EOR, but some factors remain unresolved and should be further investigated. Most importantly, a clear understanding of foam stability in the presence of oil is needed. During immiscible foam injection into a fractured, strongly water-wet core, gas propagation through the matrix was visualized by CT, albeit at zero oil saturation. *In-situ* imaging verified that gas did not enter the porous matrix at oil-wet conditions in the presence of oil, when the oil saturation varied from S_o = 0.28 to 0.76. Further investigations should be performed to disclose if gas entry to a porous matrix during foam injection is influenced by the oil phase as well as the wetting preference.

Integrated EOR

Integrated EOR efforts are increasingly important: implementation of EOR methods in a smart sequence, decreases the need for chemicals, and enhances oil recovery performance in both the near-well and far wellbore areas. In **Paper 5**, conformance control by polymer gel was an important first step in integrated EOR, to ensure an efficient recovery of oil, as well as improved microscopic and macroscopic sweep efficiencies, during chase floods. Further experiments should be performed, using polymer gel in conjunction with gas and foam chase floods at higher temperature and pressure conditions, to evaluate a polymer gel/miscible CO_2/miscible CO_2-foam sequence for use in mature reservoirs.

Foamed gels and polymer enhanced foams

Important mechanisms of polymer gel and foam floods, separately and combines, have been identified in this thesis. A logical next step is combining the two EOR methods: investigations of Polymer Enhanced Foam (PEF) or Foamed Gel (FG) applications are of interest. In PEF applications, a polymer is added to the surfactant foaming agent, while a gelant is added in FG applications. Adding a polymer (and sometimes cross-linker) to the surfactant solution improve the stability of the lamellae in foam applications. PEFs and FGs behave like foam during injection, thus low pressure gradients are potentially needed for extrusion through fractures, as shown in **Paper 4**. This concept needs to be tested experimentally.

FGs may crosslink in place, and behave similar to gel after crosslinking. Experiments must also be performed to assess the blocking ability of a Foamed Gel placed in fractures.

In general, up-scaling of existing experimental work to larger scales has potential. Polymer gel and foam experiments should, in particular, be performed on block scale, where the fracture volume constitutes a smaller part of the total pore volume than it does in experiments on core scale. Modelling and implementation in core flood and reservoir simulators are major goals.

Nomenclature

EOR	:	enhanced oil recovery
MMP	:	minimum miscibility pressure
MRI	:	magnetic resonance imaging
CT	:	computed tomography
NCS	:	Norwegian continental shelf
FV	:	fracture volume
PV	:	pore volume
k_{frac}	:	fracture permeability
b	:	fracture aperture
f_g	:	gas fraction
S_{wi}	:	irreducible water saturation
S_w	:	water saturation
S_g	:	gas saturation
S_o	:	oil saturation
OOIP	:	original oil in place
I_w	:	Amott water index
I_{AH}	:	Amott-Harvey index
CO_2	:	carbon dioxide
C_2	:	ethane
C_3	:	propane
C_4	:	butane
N_2	:	nitrogen
Cr	:	chromium
HPAM	:	partially hydrolyzed polyacrylamide
CC/AP	:	Chromium (III)-Carboxylate/Acrylamide-Polymer
US	:	the United States (of America)
T	:	temperature
P	:	pressure
ppm	:	parts per million
AFO	:	all faces open
TEO	:	two ends open
k_r	:	relative permeability

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