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Gel Dehydration by Spontaneous Imbibition of Brine from Aged Polymer Gel

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Abstract

This work investigates dehydration of polymer gel by capillary imbibition of water bound in gel into a strongly water-wet matrix. Polymer gel is a cross linked polymer solution of high water content, where water can leave the gel and propagate through porous media, whereas the large 3D polymer gel structures cannot. In fractured reservoirs, polymer gel can be used for conformance control by reducing fracture conductivity. Dehydration of polymer gel by spontaneous imbibition contributes to shrinkage of the gel, which may open parts of the initially gel filled fracture to flow and significantly reduce the pressure resistance of the gel treatment. Spontaneous imbibition of water bound in aged Cr(III)-Acetate-HPAM gel was observed and quantified. Oil saturated chalk core plugs were submerged in gel and the rate of spontaneous imbibition was measured. Two boundary conditions were tested; 1) all faces open (AFO) and 2) two ends open-oil-water (TEO-OW), where one end was in contact with the imbibing fluid (gel or brine) and the other was in contact with oil. The rate of spontaneous imbibition was significantly slower in gel compared to brine, and was highly sensitive to the ratio between matrix volume and surface open to flow, decreasing with increasing ratios. The presence of a dehydrated gel layer on the core surface lowered the rate of imbibition; continuous loss of water to the core increased the gel layer concentration and thus the barrier to flow between the core and fresh gel. Severe gel dehydration and shrinkage up to 99 % was observed in the experiments, suggesting that gel treatments may lose efficiency over time in field applications where spontaneous imbibition is a contributing recovery mechanism. The implications of gel dehydration by spontaneous imbibition, and its relevance in field applications, are discussed for both gel and gelant field treatments.

Introduction

Oil recovery from fractured reservoirs poses great challenges to the E&P industry. Fractures often exhibit permeabilities several orders of magnitude higher than the rock matrix, which may cause injected fluids to channel through fracture networks rather than displacing oil from the matrix. This can lead to early water breakthrough and low sweep efficiency. By reducing fracture conductivity, sweep efficiency and oil recovery may be significantly improved (Graue *et al.*, 2002). Injection of polymer gels to reduce flow in high permeability zones or fractures have been reported (Seright, 2004, Sydansk, 2005, Rousseau, 2005, Portwood, 1999 and 2005, Willhite and Pancake, 2008). Reducing fracture conductivity by use of gels may increase the differential pressure across matrix blocks during subsequent water or chemical floods and injected fluids can be diverted into regions that have not previously been swept. Two approaches have been studied: injection of immature gel (gelant) and injection of pre-formed gel. Gelant treatments are favorable for matrix treatments, because low viscosity and small particles allows the solution to flow through the matrix (Seright *et al.* 2003). A polymer gel is often formed by subjecting a lower viscosity gelant to a higher temperature over a certain time period, during which the solution properties change into a highly viscous and rigid gel. Injection of pre-formed polymer gel may reduce injectivity, but have been shown to have little sensitivity to physiochemical conditions in a reservoir and ensures treatment of the fractures only, as the formed gel does not penetrate significantly into porous rock (Seright, 2001, Zhang and Bai, 2011). Thus, productive oil zones will not be harmed and the chemical treatment is limited to the small fraction of the reservoir formation which constitutes the fracture volume (~1% PV). Polymer gels have an initial water content of 95 - 99.7% (Sydansk and Southwell, 2000), which may be reduced by a leakoff process that can occur when pre-formed gel propagates through fractures (Seright, 2001 and 2003). During leakoff water escapes the gel and may progress through the fracture surfaces, leaving gel in the fracture more concentrated and rigid. The pressure resistance of the gel, and thus its ability to divert chase water, increases with increasing

polymer concentration. A model for leakoff was proposed by Seright (2003), and was shown to be valid for Portland chalk during traditional leakoff experiments when the injection rate was held constant (Brattekkås, 2009).

After placement the gel may undergo processes that jeopardize its resistance to pressure. These are, for example, syneresis (Vossoughi, 2000, Zeron *et al.*, 2004) and dehydration. 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 containing gel (Dawe and Zhang, 1994, Al-Sharji *et al.*, 1999) and through bulk volumes of gel (Krishnan *et al.*, 2000). These authors suggested that imposing a pressure gradient on a bulk volume of gel after placement may dehydrate the gel. Dehydration was caused due to an imbalance of forces on either side of the gel-fluid interface, and was dependent on the rigidity of the gel.

Although gel behavior after placement in fractures has been widely studied, results are rarely discussed in conjunction with the properties of an adjacent, oil saturated, porous media. The impact relative permeability and capillary pressure in the porous matrix may have on gel behavior in fractured reservoirs is even less discussed. This work verified experimentally that an exchange of fluids between the gel and adjacent matrices may occur by spontaneous capillary imbibition of water from the gel, without imposing a pressure gradient on the system. The occurrence of spontaneous imbibition indicated that gel behavior during and after placement may be dependent on matrix properties, above all wettability. The wettability of the matrix dictates the shape of the relative permeability and capillary pressure curves, which in turn strongly influences fluid flow, and should thus be considered when planning a gel treatment and predicting its efficiency. In a strongly water-wet matrix, a positive capillary force to attract water exists. In an oil-wet matrix, water will be repelled and must overcome a capillary threshold pressure in order to invade the pores. Other matrix parameters, such as pore size distribution (rock type) and saturation will also influence the ease at which the water leaves the gel or fracture and goes on to flood the matrix.

Experiments were performed to investigate whether the capillary forces in outcrop core plugs were strong enough to extract water from formed polymer gel, and to determine the rate and end point of spontaneous imbibition in chalk. Measuring the rate of spontaneous imbibition was important as it suggest whether the spontaneous imbibition process in a gel/oil system is different from a conventional water/oil system. The gel used in the experiments was a commercially available Cr(III)-polyacrylamide. One of the differences when imbibition occurred in gel compared to brine was the immobile gel layer that formed on the rock surface as a result of gel dehydration. A thickening gel cake was observed as gel dehydrated on the core surface during imbibition, which led to an increasing skin factor that slowed the imbibition process down compared to imbibition in brine.

Experiments were also performed to determine the degree of gel shrinkage due to imbibition. A high degree of dehydration and thus shrinkage was found. This could, in time, open large parts of the gel-treated fractures to flow and decrease the efficiency of the gel treatment. According to numerous authors, gel treatments have in many field applications been less effective than expected in reducing water production from fractured wells, or the treatments have lost efficiency over time (White *et al.*, 1973, Seright, 2003, Portwood, 2005). This study may contribute to new understanding of gel failure after treatment in water-wet fields.

Experiments

The experimental schedule was two-fold;

- a) a set of experiments were conducted to investigate spontaneous imbibition rates from cores fully submerged in gel. Two boundary conditions for spontaneous imbibition were tested; 1) all faces open (AFO) and 2) two ends open (TEO-OW), where one end face was in contact with water or gel and the other end face was in contact with oil. Core plugs and corresponding boundary condition are listed in **Table 1**.
- b) a set of experiments were conducted to quantify shrinkage of gel driven by capillary spontaneous imbibition.

Different experimental setups were required and are outlined below.

Core material and preparation

Chalk blocks were obtained from the Portland Cement Factory in Aalborg, Denmark, and cylindrical core samples of various diameters and lengths were drilled out. 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%). The brine permeability and porosity ranges from 1-10 mD and 43-48%, respectively. More details about the rock may be found in Ekdale and Bromley (1993), and Hjuler (2007). Edwards limestone from a quarry in west Texas, USA was used in an introductory experiment. Trimodal pore sizes, vugs and microporosity have been identified using thin section images, mercury injection and NMR T2 relaxation experiments. The brine permeability and porosity ranges from 3-28 mD and 16-24% (Tipura (2008) and Riskedal (2008)). Bentheim sandstone from the Gildehausen quarry near Bentheim, Germany was also used. The Bentheim sandstone is homogeneous in terms of porosity and permeability, averaging at 23% and 1100 mD, respectively, with mean composition of 95% quartz, 3% kaolinite, and 2% orthoclase. More information may be found in Klein and Reuschlè (2003) and Schütjens (1995).

A total of 24 core plugs were prepared for this study. All cores were washed and dried at 80°C for several days before they were vacuum evacuated and saturated directly with oil. The porosity and pore volume of each core plug was determined

from weight measurements. Cores used for TEO-OW boundary condition experiments and gel shrinkage experiments were coated with epoxy along the core length so only the end faces were open to flow, and a POM (Polyoxymethylene) end piece was fitted at one end of the core. Cores used in gel shrinkage experiments were fitted with a POM end piece at the outlet end and a plastic container at the inlet, to hold a specified gel volume. After saturation, the cores were subjected to spontaneous imbibition tests in brine or pre-formed polymer gel. All experiments were performed at ambient temperature. An overview of all cores and experiments is shown in **Table 1** and core properties may be found in **Table 2**.

Table 1: Core overview

Core ID	Comment	Experiment type	Boundary condition	Saturated with
1 SI test	P. chalk	SI in gel	AFO	Decane
2 SI test	B. sandstone	SI in gel	AFO	Decane
3 SI test	E. Limestone	SI in gel	AFO	Decane
1_SI		SI in brine	AFO	Lamp oil
2_SI		SI in brine	AFO	Lamp oil
3_SI		SI in gel	AFO	Lamp oil
4_SI		SI in gel	AFO	Lamp oil
5_SI		SI in gel	AFO	Lamp oil
6_SI		SI in gel	AFO	Lamp oil
7_SI		SI in gel	AFO	Lamp oil
1_CC		SI in brine	TEO-OW	Lamp oil
2_CC		SI in brine	TEO-OW	Lamp oil
3_CC		SI in brine	TEO-OW	Lamp oil
4_CC		SI in gel	TEO-OW	Lamp oil
5_CC		SI in gel	TEO-OW	Lamp oil
6_CC		SI in gel	TEO-OW	Lamp oil
7_CC		SI in gel	TEO-OW	Lamp oil
8_CC		SI in gel	TEO-OW	Lamp oil
1_GS		Gel shrinkage	TEO (vertical)	Decane
2_GS	*	Gel shrinkage	TEO (vertical)	Decane
3_GS	**	Gel shrinkage	TEO (vertical)	Decane
4_GS	**	Gel shrinkage	TEO (vertical)	Decane
5_GS		Gel shrinkage	TEO (vertical)	Decane
6_GS		Gel shrinkage	TEO (vertical)	Decane

* imbibition stopped after first batch of gel, before the end point

** several batches of gel added for imbibition

Fluids

The gel used was 0.5 % HPAM (Ciba Alcoflood 935, ~5 million daltons molecular weight) cross-linked by 0.0417 % Cr(III)-acetate and aged at 41°C for 24 hours (five times the gelation time). After aging, the gel was cooled to ambient temperature before initiation of imbibition experiments. The fluid properties of all used fluids are given in **Table 3**.

Spontaneous imbibition

Initial experiments were performed using three outcrop core materials of different capillarity: 1 SI test, 2 SI test and 3 SI test (**Table 1** and **Table 2**). The cores were saturated with oil and dropped in beakers containing aged gel. The induction time was recorded, and the cores were left in the beakers until spontaneous imbibition ended. At this point, the cores were weighed and recovery calculated from material balance equations. The rates of oil recovery and spontaneous imbibition were not measured for these cores, and the end points were estimated from visual observations. For cores 1_SI through 7_SI and 1_CC through 8_CC, imbibition rates were determined during spontaneous imbibition with cores fully submerged in either brine or gel using one of two boundary conditions:

1) All Faces Open (AFO) boundary conditions: the rate of spontaneous imbibition versus time was quantified by gravimetric measurements of the core sample and brine or gel respectively, using the setup showed in **Figure 1A**). The core and fluid weights were both logged versus time. At given time steps, the core was taken out of the brine or gel bath, excess fluid (oil drops and gel) was carefully wiped off the surface and the core was weighed. This approach was used because a steadily

increasing gel film occurred on the core surface during spontaneous imbibition in gel, which captured the displaced oil. After weighing, the core was re-submerged in the fluid. The displaced oil volume, and hence the rate of recovery was calculated at each time step using material balance equations. The core end point saturation was determined from weight measurements after ended imbibition. The gel layer on the core surface was also collected and weighed. A thin layer of oil on top of the polymer gel or brine prevented evaporation of water during long term tests.

2) Two Ends Open- Oil-Water (TEO-OW) boundary conditions were also used. The experimental setup, shown in **Figure 1B**), ensured that one end face of the core was in contact with the aqueous, imbibing, phase and the other with the oleic phase: zero capillary pressure is obtained at the second end face. Due to capillary pressure being present only at the inlet end face, displacement of oil mainly occurs at the outlet end of the core when the core is aligned horizontally, except for a moment right after imbibition initiation. This makes it possible to quantify the bulk of the oil production directly by volume recordings, also during imbibition in gel. After imbibition in gel ceased, the dehydrated gel layer was removed from the inlet end face and all cores were submerged in brine for further spontaneous imbibition.

Table 2: Core properties

Core ID	Length [cm]	Diameter [cm]	Area open for imbibition [cm ²]	Volume/ open surface ratio [cm]	Pore volume [ml]	Porosity [%]	S _o endpoint to gel [%]	S _o endpoint to water [%]
1 SI test	9.1	3.80	131.32	0.79	45.53	44.12	68.3	68.3
2 SI test	5.92	3.80	93.36	0.72	14.27	21.26	66.97	66.97
3 SI test	8.84	3.80	128.21	0.78	16.6	16.56	52.84	52.84
1_SI	6.04	5.09	137.28	0.90	53.76	43.74	-	62.13
2_SI	6.04	5.07	136.58	0.89	54.05	44.33	-	62.53
3_SI	7.97	5.09	168.04	0.96	75.11	46.36	65.11	65.11
4_SI	7.92	5.09	167.26	0.96	75.92	47.14	59.48	59.48
5_SI	7.92	5.07	166.59	0.96	77.81	48.63	57.41	57.41
6_SI	8.01	5.10	169.19	0.97	77.28	47.23	56.34	56.34
7_SI	8.02	5.07	167.91	0.96	75.92	46.98	60.54	60.54
1_CC	6.00	3.79	11.28	6.00	33.42	49.37	-	61.64
2_CC	6.00	3.82	11.46	6.00	33.14	48.19	-	57.64
3_CC	10.51	3.79	11.28	10.51	56.03	47.25	-	57.12
4_CC	6.00	3.80	11.34	6.00	33.67	49.48	44.03	54.3
5_CC	10.30	3.80	11.34	10.30	56.57	48.17	47.18	52.27
6_CC	6.00	3.80	11.34	6.00	27.34	40.17	43.16	43.16
7_CC	10.30	3.80	11.34	10.30	47.06	40.28	24.01	24.01
8_CC	10.45	3.80	11.34	10.45	51.76	43.67	22.21	22.21
1_GS	5.23	3.72	10.87	5.23	26.71	46.99	64.69	-
2_GS	6.89	3.74	10.96	6.89	34.88	46.20	28.32	-
3_GS	12.62	3.65	10.43	12.62	56.21	42.68	53.04	63.00
4_GS	11.85	3.61	10.24	11.85	52.96	43.66	60.76	64.91
5_GS	6.01	3.88	11.82	6.01	32.23	45.36	56.57	56.57
6_GS	6.03	3.84	11.58	6.03	31.54	44.24	58.97	59.76

Gel Shrinkage

One of the most important applications of spontaneous imbibition of water from gel is gel shrinkage; blocking of fractures by gel is most efficient if the entire fracture volume, or significant parts of it, is filled with low permeability gel. Gel shrinkage is in this case defined as the percentage of initial gel volume that is lost due to spontaneous capillary imbibition of water from the gel. Shrinkage of gel during spontaneous imbibition was measured by exposing an oil saturated core plug to a limited volume of gel and recording the reduction in gel volume during spontaneous imbibition. The setup was similar to TEO-OW, but the cores were placed vertically with a specified gel volume on top. This ensured contact between the gel and rock surface at all times. The shrinkage of gel by spontaneous imbibition was recorded directly by volume measurements. At the end point, the gel film was collected and weighed to determine total gel shrinkage. If the end point for spontaneous imbibition was not reached using one batch of gel, one or several more batches of gel could be added to the plastic container. After removal of the

final gel layer, brine was placed in the container to ensure that the potential end point for imbibition was reached, or to define the final end point. A schematic of the setup is showed in **Figure 1C**).

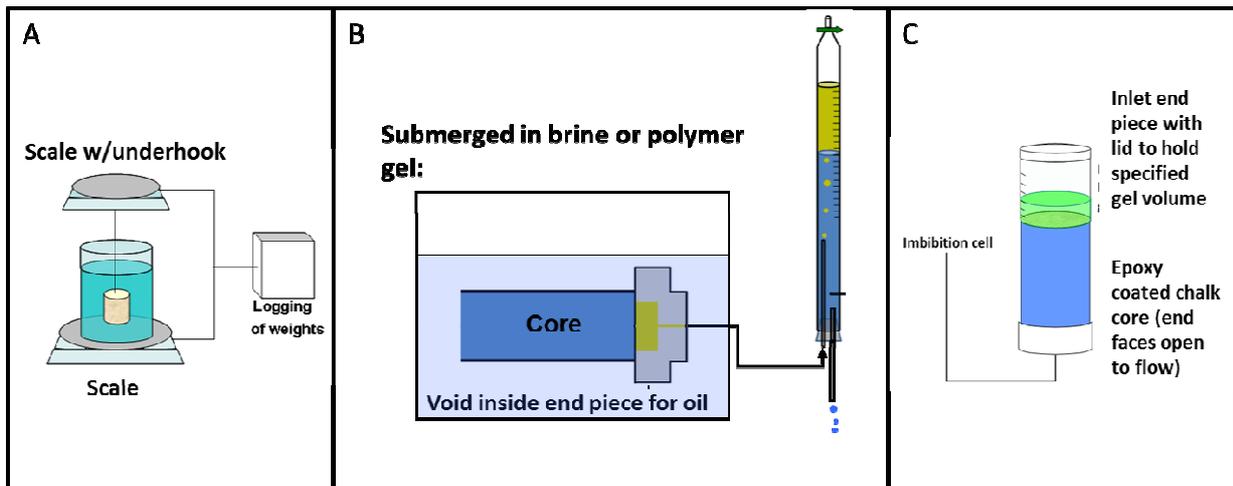


Figure 1: A) Schematic setup for continuous measurements of gel and core weight versus time using AFO boundary conditions. B) Schematic setup for co-current imbibition using TEO-OW boundary conditions. C) Schematic setup for gel shrinkage experiments.

Table 3: Fluid properties

Fluid	Density [g/cm ³]	Viscosity [10 ⁻³ Pa·s] at 20°C	Composition
Ekofisk brine	1.048	1.09	4.00 Wt-% NaCl 3.40 Wt-% CaCl ₂ 0.50 Wt-% MgCl ₂ 0.05 Wt-% NaN ₃
n-Decane	0.73	0.92	-
Refined lamp oil	0.74	1.43	-
HPAM Gel	~1.048	~2*10 ⁶ after gelation*	0.5% HPAM 0.0417% Cr(III)-Acetate Ekofisk brine

* measured at 0.001 1/s shear rate after 24 hours of gelation (Liu and Seright, 2001)

Results and Applications

Spontaneous imbibition

Spontaneous imbibition of water from pre-formed gel was first observed in three cores, 1 SI test (chalk), 2 SI test (sandstone) and 3 SI test (limestone) that were fully saturated with oil and placed in beakers of aged polymer gel. The spontaneous imbibition process dehydrated gel close to the core surface and continued until the end point for imbibition was reached in the cores, regardless of capillarity. The induction time was, however, influenced by capillarity and was shortest in chalk (1.5 minutes) compared to Edwards limestone (11 minutes) and Bentheim sandstone (53 minutes). The rate of imbibition was not measured in the first three experiments, but the procedure was extended in the next fifteen core plugs, 1_SI through 7_SI using AFO boundary conditions and 1_CC through 8_CC using TEO boundary conditions, to make these measurements possible. Visual recordings were challenging for AFO cores, as displaced oil volumes were trapped in the gel and could not be separated from it and accurately measured neither during spontaneous imbibition nor after the process ended. Weight measurements were therefore used for monitoring the development in average oil and water saturations during imbibition. During spontaneous imbibition, gel dehydrates on the core surface and forms a filter cake of more concentrated gel. This filter cake influences the accuracy of the gravimetric measurements; the layer of dehydrated gel will add to the core weight and overestimate the recovery if it is not accounted for. Intuitively, the amount of dehydrated gel on the core surface will depend on the amount of water imbibed. This means that the immobile gel layer will increase faster when the rate of spontaneous imbibition is higher. The development of the gel layer versus time, quantified by the weight of dehydrated gel, will thus mimic the spontaneous imbibition curve and may be determined using Eq. [1]:

$$m_{gf}(t) = \frac{\Delta m(t)}{\Delta m_{max}} \cdot m_{gf,max} = \frac{(m_{(c+gf)}(t) - m_i)}{(m_{(c+gf),max} - m_i)} \cdot m_{gf,max} \quad [1]$$

where $m_{gf}(t)$ is the gel film weight as a function of time, $\Delta m(t)$ is the difference between the initial weight m_i and the weight of the core (including gel film) as a function of time, $m_{(c+gf)}(t)$. Δm_{max} is the maximum weight change; the difference between initial and endpoint core weights (including the gel film), $m_{(c+gf),max}$. $m_{gf,max}$ is the gel film weight at the endpoint of spontaneous imbibition, and is found by weighing the core with and without the gel layer. The removal of the gel layer must be done cautiously, as the material balance calculations depend on the core being intact throughout the experiment. **Figure 2** shows core 1 SI Test during imbibition, and illustrates the oil drops that were trapped by gel on the core surface or in the core vicinity.

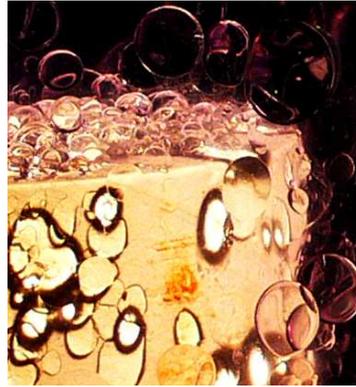


Figure 2: Oil adhesion to the core surface during imbibition

Figure 3 shows the recovery of oil during spontaneous imbibition for cores 1_SI through 7_SI, scaled in accordance to Ma *et al.* (1997). The black lines represent the spontaneous imbibition curves for brine, while the grey lines show the imbibition curves for cores submerged in gel. The imbibition in gel was a factor ten slower than imbibition in brine. The endpoint for spontaneous imbibition, equal to the residual oil saturation for this particular core material (Viksund, 1998), was reached in all experiments, also when the cores were submerged in gel. The endpoint was confirmed by placing the cores in brine for further spontaneous imbibition after removal of the dehydrated gel layer.

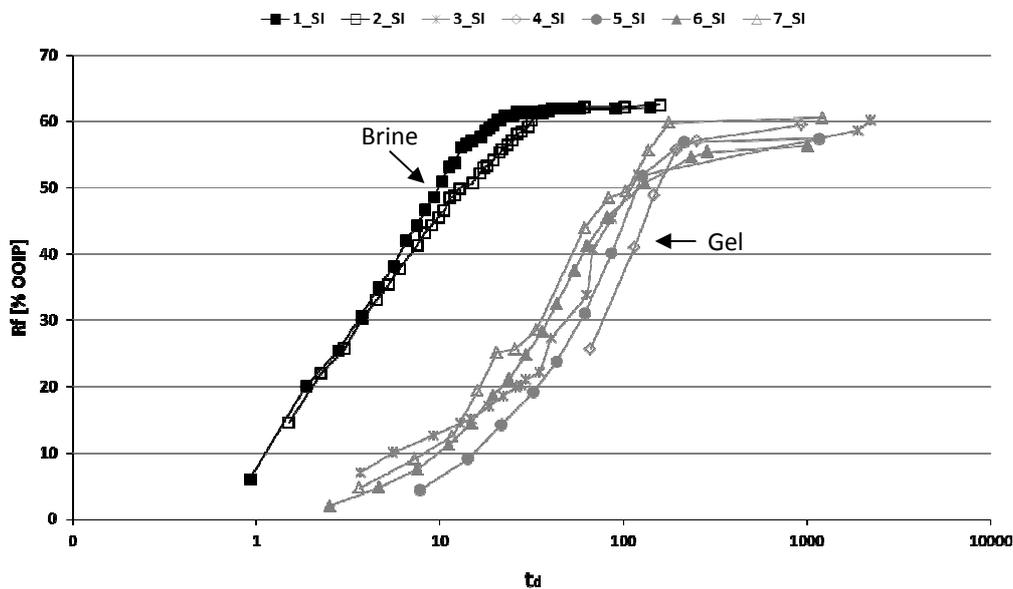


Figure 3: Oil recovery during spontaneous imbibition.

Every marker in **Figure 3** represents a point in time where the cores have been taken out of the brine or gel bath and weighed in air. Before weighing, excess gel and oil drops were removed from the core surfaces, but the dehydrated gel film was left unharmed. Weight recordings started at 4_SI; $t = 91$ minutes, 5_SI; $t = 10$ minutes, and 6_SI and 7_SI weights were recorded $t = 2$ minutes after initially submerging the cores in gel. Both oil recovery and the rate of spontaneous imbibition were lower at a given time when the first weight recording was later in the experiment. During spontaneous imbibition in gel, 4_SI therefore

had the lowest rate of recovery, 5_SI had a slightly higher rate, and 6_SI and 7_SI had the highest rates of recovery and imbibition. The reason for this behavior may be the filter cake formation on the core surfaces during imbibition; shortly after immersion in aged gel, the dehydrated gel layer is thin and may be partially removed together with the oil drops and excess gel at weighing, which will make the core surface more easily accessible for further spontaneous imbibition. This was confirmed by partially removing gel from core 7_SI at time steps three, four, ten and eleven, which resulted in a more rapid spontaneous imbibition in the core and higher recovery values at the subsequent time steps. The rate of recovery was comparable to 6_SI when the dehydrated gel layer was left undamaged. Core 3_SI was not weighed in air, but left in the container for the whole duration of the experiment. Consequently, oil drop adhesion to the core surface was not corrected for and oil recovery was overestimated early in the experiment. Large scale disturbances were experienced in the data set due to oil drops growing and letting go of the core surface, thus 3_SI was omitted when calculating imbibition rates. **Figure 4A)** shows the imbibition rates in both water and gel and **Figure 4B)** shows the imbibition rates in gel only. The imbibition rates were severely reduced when cores were submerged in gel instead of brine, although the induction time was the same: oil production was observed approximately 90 seconds after core immersion in both gel and brine.

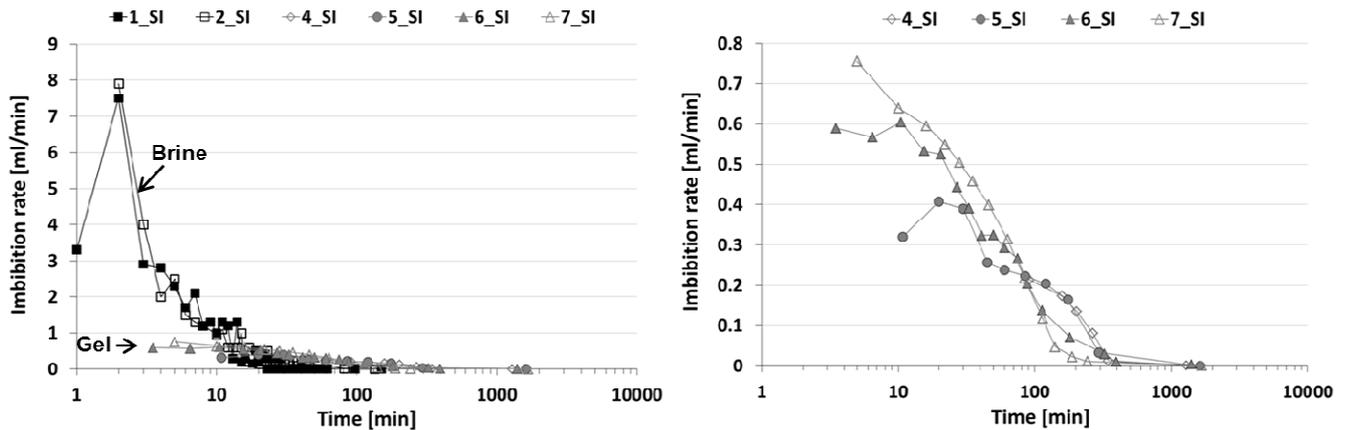


Figure 4: A) The rate of spontaneous imbibition in brine and gel. B) The rate of spontaneous imbibition in aged polymer gel alone.

To enable visual recordings, and thus enhance the accuracy of the measurements, cores with TEO-OW boundary conditions were prepared. During spontaneous imbibition experiments in brine using these boundary conditions, minor amounts of oil, (corresponding to 2 – 4 % of the total core pore volume) were produced at the inlet end (Haugen *et al.*, to be published). Direct recordings of oil production at the outlet end therefore reflected the imbibition process in the core, also when the fluid contacting the inlet end face was gel. Cores 1_CC, 2_CC and 3_CC were subjected to co-current spontaneous imbibition tests in brine, while 4_CC, 5_CC, 6_CC, 7_CC and 8_CC of similar dimensions were subjected to gel testing. No change in induction time was observed between water and gel imbibition and oil was produced shortly after immersion. The production curves from co-current imbibition using TEO-OW boundary conditions are shown in **Figure 5**.

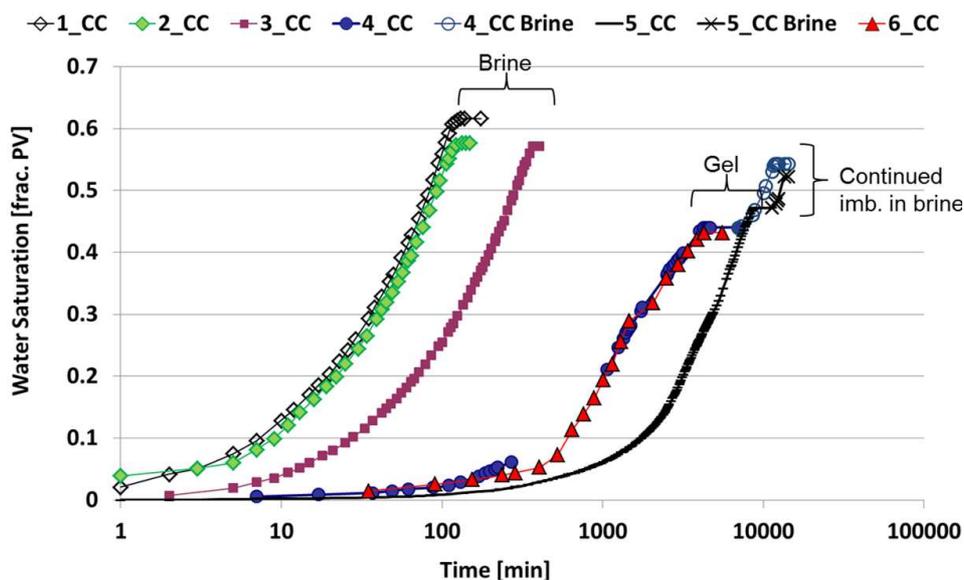


Figure 5: Recovery during co-current imbibition for cores of different lengths (and thus different bulk volume to open surface ratios) submerged in brine or gel.

Figure 6A) shows the imbibition curves as a function of the square root of time for the shorter cores (6 cm) and **Figure 6B)** for the longer cores (10 cm). Oil recovery as a function of square root of time was not linear in gel/oil systems. The co-current imbibition process was slow in both brine and gel, and the residual oil saturation was reached after a long period of time when the cores were submerged in brine. During gel testing the endpoint for spontaneous imbibition was not reached, and continued imbibition in brine was possible after gel testing when the filter cake had been removed from the inlet end face. For a short time span after immersion in brine or gel, counter-current production of oil was observed. In most cores, counter-current production was minor and the change to pure co-current production was swift. Some cores, however, produced a lot of oil counter-currently. Examples are core 7_CC and 8_CC, which produced much of the oil at the inlet end and imbibition curves could not be generated. Mason *et al.* (2009) also reported a larger scatter in results when the area open to imbibition decrease. Oil produced counter-currently was trapped in the gel at the inlet and could therefore not be quantified gravimetrically or volumetrically.

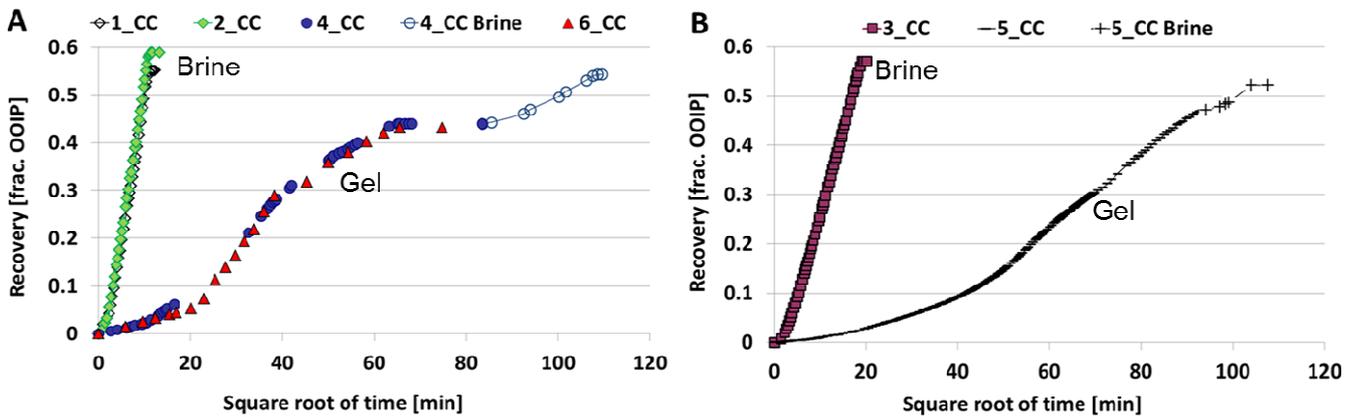


Figure 6: A) Recovery versus the square root of time for 6 cm cores. B) Recovery versus the square root of time for 10cm cores.

A recovery rate dependency on bulk volume to open surface ratio was detected during immersion in both brine and gel. The bulk volume to open surface ratio (here termed VSR) is defined as

$$VSR = V_b/A_{surf} \quad [2]$$

where V_b is the bulk volume of the core sample [cm^3] and A_{surf} is the surface area open to flow [cm^2]. Increasing VSR led to lower recovery rates. The delay caused by increasing VSR values may be corrected by accounting for the dimensional differences: dividing imbibition time by core length squared (Haugen *et al.*). The scaled results are shown in **Figure 7**. The delay seen between the brine and gel imbibition curves must be caused by interactions between the core surface and the polymer gel, particularly the thickening filter cake on the core surface: gel particles will not enter the small chalk pores, thus the two-phase flow within each core during imbibition constitutes brine and oil only.

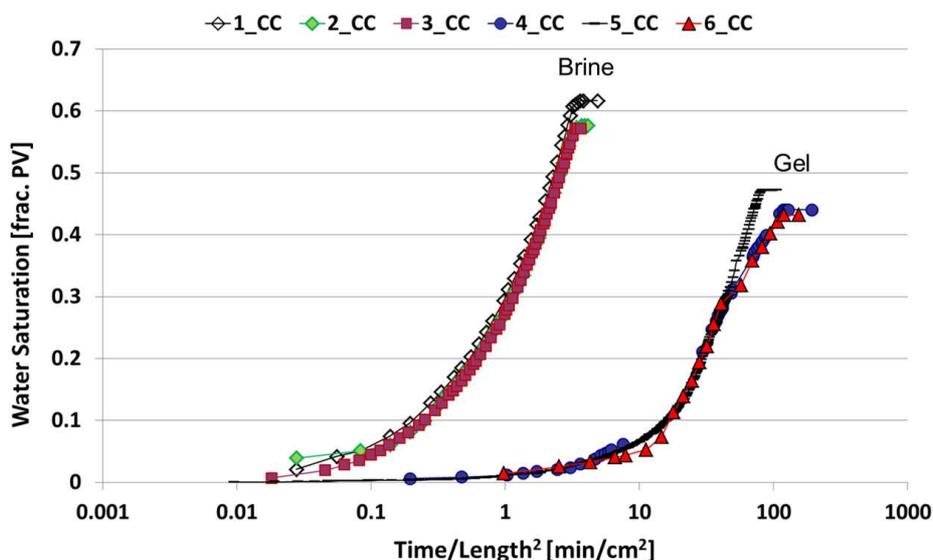


Figure 7: Recovery during imbibition versus $\text{Time}/\text{Length}^2$. The dimensional differences are accounted for by dividing imbibition time by core length squared, thus the time difference between the curves is due to the polymer gel characteristics or core surface/polymer gel interactions.

The filter cake that forms on the core surface introduces a skin factor, that increases during imbibition as the thickness and concentration of the filter cake grows. The endpoint for spontaneous imbibition was not reached during imbibition in gel as capillary forces were counter-acted by the skin factor thus stopping the imbibition process. When imbibition stopped, the dehydrated gel layer which constitutes the filter cake was removed from the end face and the cores were immersed in brine for further imbibition to reach the final endpoint.

The rates of imbibition for TEO-OW boundary condition experiments are shown in **Figure 8**. During imbibition in brine, slightly increased initial values were seen, after which the rates slowly decreased. Imbibition ceased at the residual oil saturation. Dimensional differences did not influence the imbibition rate, and the longer cores experience the same rates as the shorter cores, though they continue to imbibe water for an extended period of time. During TEO-OW imbibition in gel, the imbibition rates were low, peaking at approximately 0.03 ml/min. At the end point, when the filter cake was removed and the cores were placed in brine, the imbibition rates decreased further (~ 0.001 ml/min). Few visual recordings and slight changes in displaced oil volume per time step makes it difficult to quantify the changes in imbibition rate with tolerable uncertainty. Exact values or graphs will for this reason not be given.

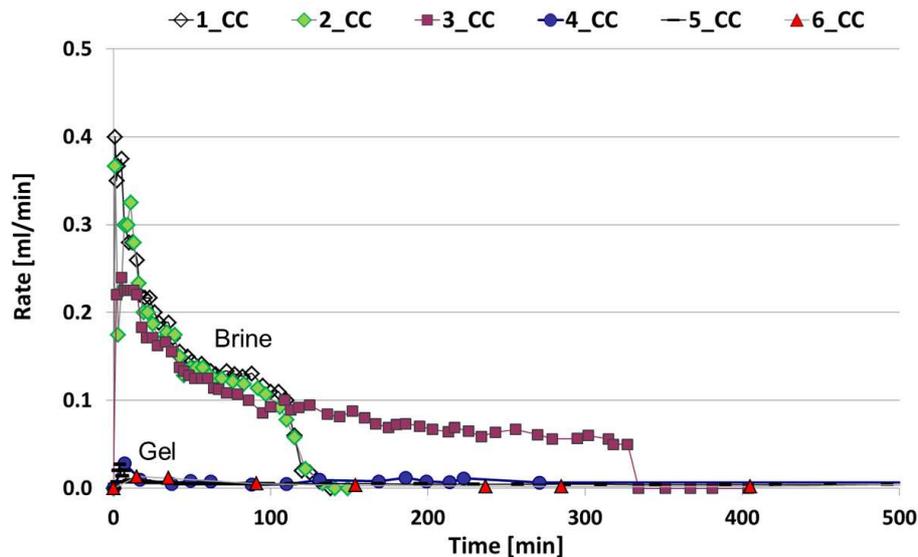


Figure 8: Rate of co-current spontaneous imbibition in cores of different lengths and TEO boundary conditions submerged in brine or gel. The x-axis is in this case limited to the time where brine imbibition still occurs.

During co-current imbibition, the cores submerged in gel required approximately 30 times longer to reach their respective end points, which were lower than the recorded end points for imbibition in brine. This factor is expected to vary based upon the capillarity of the rock, the gel concentration and the water/oil mobility ratio, and was also shown to vary with VSR. **Figure 9** shows the additional time needed to obtain a given water saturation during imbibition in gel compared to brine for AFO cores (VSR ~ 1 cm) and TEO cores of different VSR (~ 6 cm and ~ 10 cm). The curves in **Figure 9** were constructed by subtracting the imbibition time in water from the imbibition time in gel at given water saturation points for each VSR. Cores with lower VSR achieve high water saturations faster during gel injection than cores of higher ratios. This implies that spontaneous imbibition of gel-bound water may be faster and more severe in highly fractured reservoirs, where VSR is lower.

Gel shrinkage

Six cores were prepared with TEO boundary conditions to measure gel shrinkage, and imbibition experiments were performed with a limited volume of gel available for imbibition. Shrinkage of gel due to dehydration during spontaneous imbibition was recorded. The available gel volume varied from 0.03 – 1 PV. **Table 4** shows data for gel shrinkage experiments, including available gel volume in milliliters, fraction of pore volume and fraction of oil saturation. Initial gel film thickness and volumetric shrinkage after imbibition is also tabulated. Core 3_GS and 4_GS had four consecutive placements of a known volume of gel, and each placement of gel is tabulated.

Figure 10 shows shrinkage of the gel film as function of time for each core and gel placement. Because the cores were placed vertically with a gel volume of top, gravitational effects could not be neglected and oil production occurred at the inlet as well as the outlet end, especially at low water saturations. Collection and recording of the oil produced at the inlet end was possible due to the limited amount of gel present, but in many cases a fraction of the oil was bound in the gel and could not be collected until the end point for imbibition was reached. All counter-currently produced oil could be collected when the gel film was removed from the core surface, as oil was not dispersed in the gel but merely trapped in it. Some of the curves in **Figure 10** show a sudden increase in gel shrinkage between the two last points due to this: the shrinkage curves of cores 3_GS, 3rd and 4th gel and 4_GS, 3rd and 4th gel exemplifies this behavior.

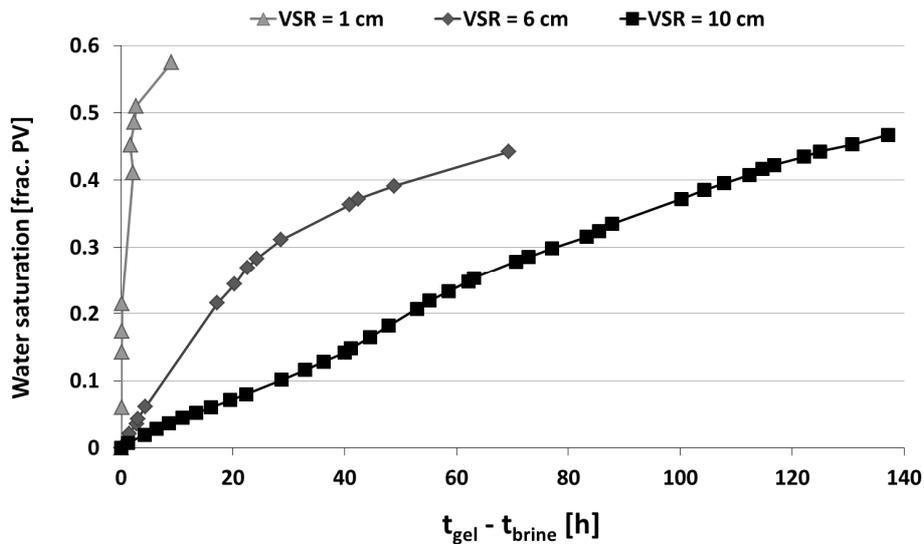


Figure 9: The additional time it takes to imbibe water during imbibition in gel compared to brine increase with water saturation and increasing VSR.

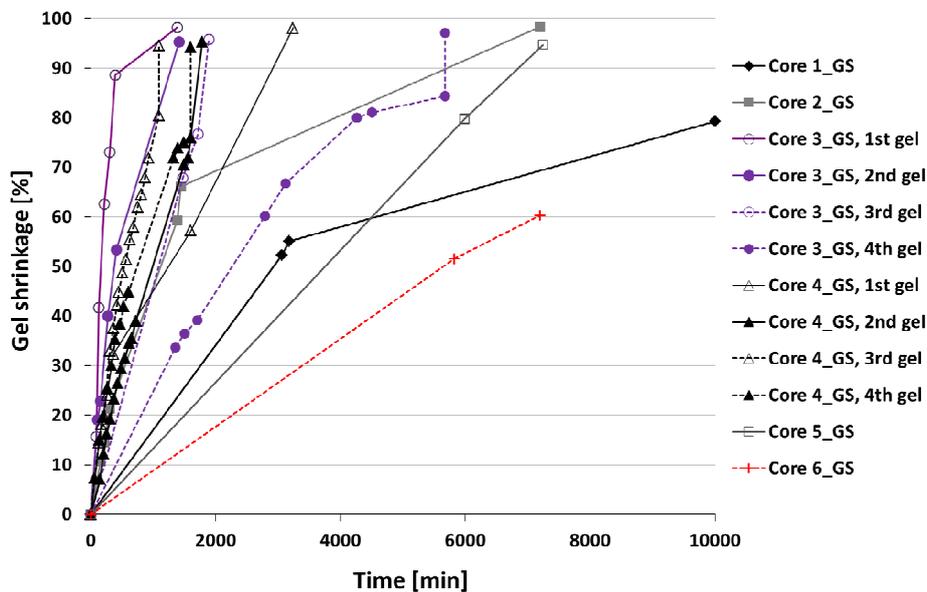


Figure 10: Gel shrinkage as a function of time for SI cores

Figure 11 shows the gel shrinkage curves as functions of the square root of time. Cores with few data points were not included. The core with the highest available gel volume in fraction of PV (6_GS) exhibited the slowest gel shrinkage. Following 6_GS (available gel volume, PV = 0.98) was 1_GS (PV available = 0.82), and 3_GS 4th gel placement (0.4 PV). 2_GS (0.30 PV), 4_GS 4th gel placement (0.29 PV) and 4_GS 2nd gel placement (0.21 PV) showed comparable shrinkage trends. 4_GS 3rd gel placement (0.20 PV) was similar to core 4_GS 4th gel placement up to $t^{1/2} = 30$, but exhibited a higher imbibition rate after this time. 3_GS 1st gel placement (0.03 PV) and 3_GS 2nd gel placement (0.1 PV) had the smallest available volumes of gel, and exhibited the fastest gel shrinkage. This behavior is expected: a 10% increase in core water saturation corresponds to a higher degree of gel shrinkage if the available gel volume is low.

Figure 12A) shows the relationship between normalized oil recovery and gel shrinkage: if the available gel volume is less than the potential volume for spontaneous imbibition, gel shrinkage is fairly predictable for this rock-oil-gel system. For the two cores where the available gel volumes exceeded the imbibition potential, however, the gel shrinkage decreased. Figure 12B) shows the degree of gel shrinkage plotted against the gel volume available for spontaneous imbibition. The curve is a third degree polynomial fit to the experimental data and shows that the gel will shrink approximately 98% when the available gel volume is less than 0.6 of the matrix pore volume, which also correspond to the end point for spontaneous imbibition for Portland chalk. At ~0.6 PV there is a break point where shrinkage is sensitive to the amount of gel available. The break point will probably be dependent on the end point for spontaneous imbibition, which varies between different rock types, but is fairly stable for this core material. In fractured reservoirs, ~1 % of the pore volume is typically held in fractures; this implies

that the volume of available gel in fractures usually is very low compared to the matrix pore volume, and a fast shrinkage process may be expected due to this.

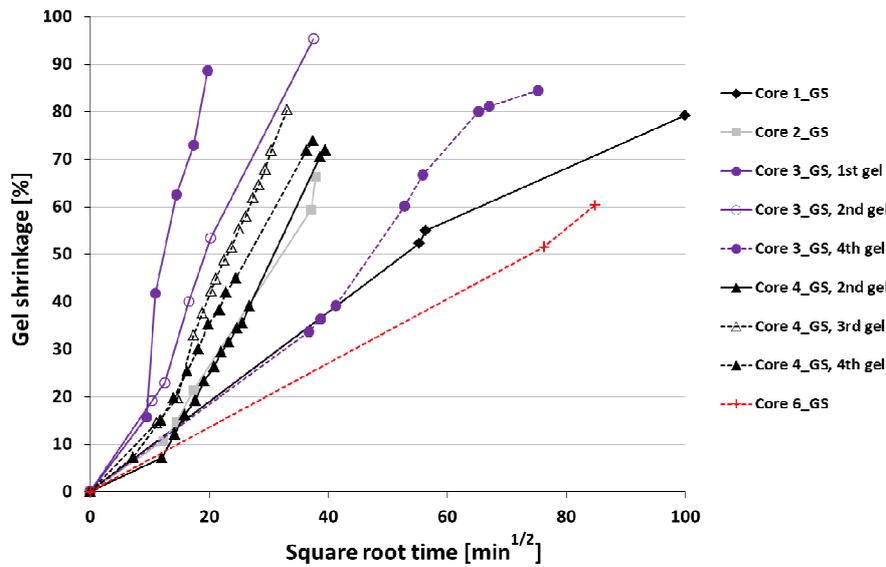


Figure 11: Gel shrinkage as a function of square root of time for some cores

Table 4: Gel shrinkage experiment specifics.

Core ID	Gel placement [#]	Available gel			Initial gel film thickness [cm]	Shrinkage [%]
		[ml]	[frac PV]	[frac So]		
1_GS	1 st	21.81	0.82	0.82	2.01	79.22
2_GS	1 st	10.28	0.29	0.29	0.94	98.28
3_GS	1st	1.92	0.03	0.03	0.18	99.46
	2nd	5.25	0.09	0.10	0.50	99.46
	3rd	7.96	0.14	0.16	0.76	95.80
	4th	18.13	0.32	0.44	1.74	97.16
	Total	33.25	0.59	-		
4_GS	1st	6.64	0.13	0.13	0.65	98.13
	2nd	9.87	0.19	0.21	0.96	95.26
	3rd	7.59	0.14	0.20	0.74	94.59
	4th	9.65	0.18	0.29	0.94	94.16
	Total	33.74	0.64	-		
5_GS	1st	19.27	0.60	0.60	1.63	94.65
6_GS	1st	30.84	0.98	0.98	2.61	60.31

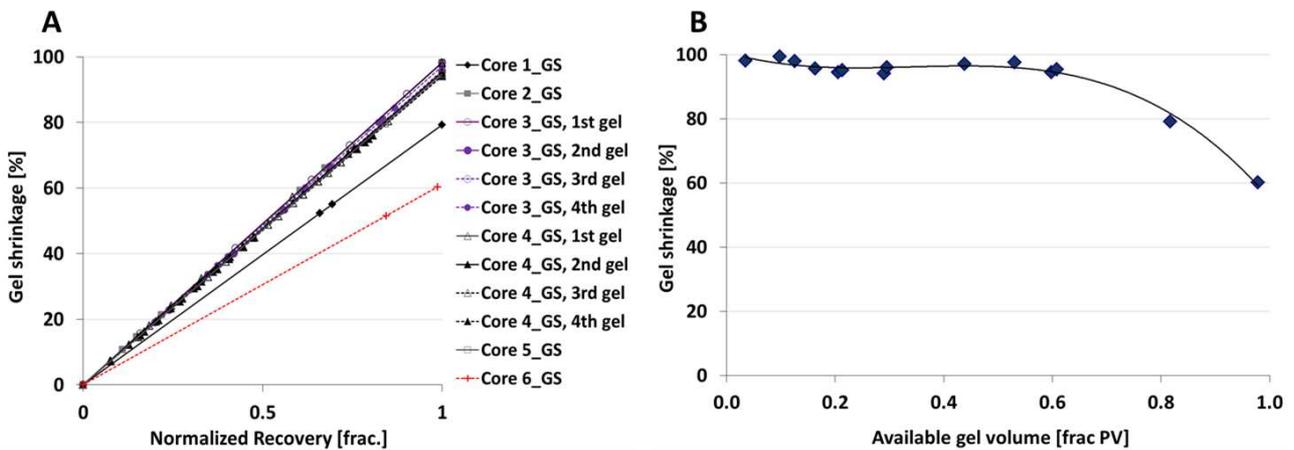


Figure 12: A) Gel shrinkage as a function of normalized recovery. B) Gel shrinkage as a function of available gel volume [PV].

Figure 13 shows pictures of the shrinkage process in 3_GS 4th gel placement at four different time steps during imbibition. The gel changes colour during shrinkage; a darkening of colour means that the chrome and polymer concentrations in the gel film are increasing. The degree of shrinkage in this case was 97%, meaning that an initial average gel film thickness of 1.74 cm has shrunk to 0.05 cm due to spontaneous imbibition.

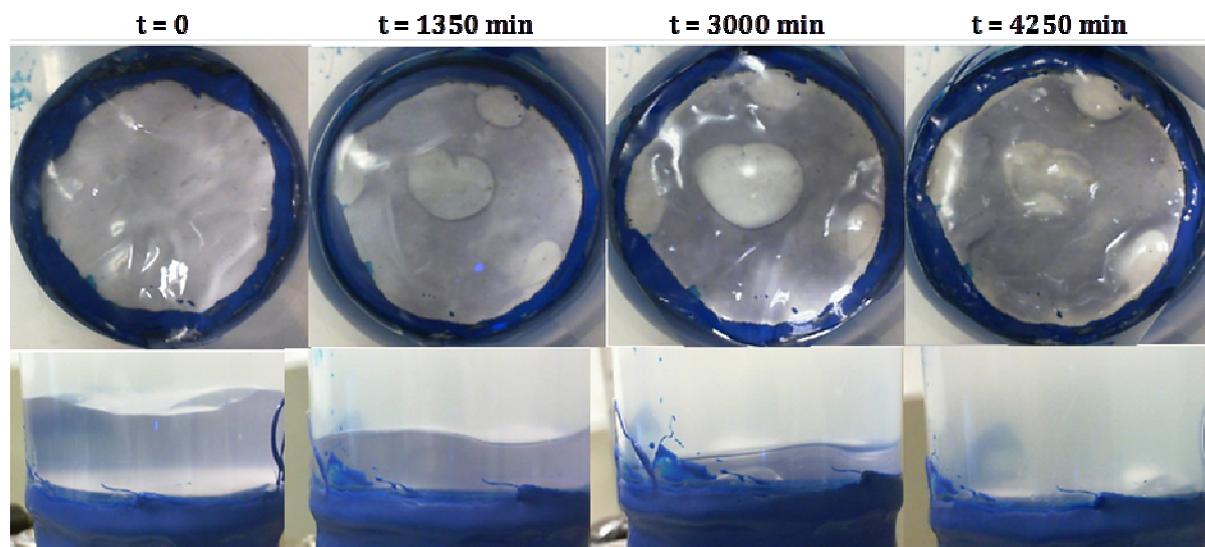


Figure 13: Core 3_GS, 4th gel placement. Shrinkage: 97.16 %. Trapped oil drops are observed in the images. Top row: core seen from bird view. Bottom row: shrinking gel layer seen from the side.

The concentration of the polymer gel layers after imbibition was estimated for AFO cores using oil recovery and the weight of the collected gel film. The gel film was successfully collected with consistent results in all five AFO gel experiments. To simplify the calculations, two assumptions were made: 1) all dehydrated gel was removed from the surface and the weight of the finite gel cake was thus correctly quantified, 2) no radial gradient of gel layer concentration exists, meaning that the collected gel had a uniform concentration. Both assumptions are dependent on the immobility of all dehydrated gel (Seright, 1998). The results are shown in **Figure 14A**). The concentration of the collected gel films after spontaneous imbibition was estimated to be approximately 4.2 times above the original polymer gel concentration for AFO boundary conditions. For gel shrinkage experiments, the final concentrations were dependent on the available gel volume, and are shown in **Figure 14B**) The maximum gel concentration achieved in gel shrinkage experiments ($C/C_0 \approx 60$) coincides with the maximum concentration reported during extrusion through fractures in earlier work (Seright, 2001). This value may represent a maximum achievable gel concentration at moderate pressure conditions, and may be associated with the osmotic pressure in the highly concentrated gel which counteracts further dehydration by capillary forces. For TEO experiments, the gel films were not successfully collected and the gel concentrations therefore not estimated.

The spontaneous imbibition of brine from gel is significant on core scale at strongly water-wet conditions. On reservoir scale, the gel treated area may have less water-wet or even oil-wet conditions, in which case the capillary forces will either be less dominant or completely inhibit imbibition of brine from gel. An increased initial gel concentration in the fracture, due for example to leakoff, may also slow the imbibition of brine from gel because the gel will be stronger. The impact of gel shrinkage on fracture conductivity was calculated using the cubic law of Witherspoon *et al.* (1980), where the absolute permeability of the fracture is a function of fracture aperture (b) only:

$$K_{abs} = b^2/12 \quad [2]$$

, and the relationship between gel permeability (k_{gel}) and polymer concentration (C) given by Seright (2006):

$$K_{gel} (\mu D) = 125 C^{-3} \quad [3]$$

Figure 15 shows fracture conductivity calculated as a function of gel shrinkage. Fracture apertures of 0.001 mm to 10 mm were investigated and the fractures were assumed to be initially 100 % gel filled. The initial polymer concentrations were estimated to 0.5% (solid lines) and 5% (dotted lines) respectively, to reflect gel of typical injected composition and gel that has experienced leakoff and thus gained a higher concentration. The calculations estimated that 1% gel shrinkage will increase the conductivity of the largest fracture (10 mm) by 10 000 mD and the conductivity of the 1 mm fracture by 100 mD, while fractures < 1 mm will experience a conductivity increase of <<10 mD. After the gel has shrunk 10 %, the large fractures will have conductivities 10^7 and 10^5 mD higher than the initial fracture permeability, respectively, and tighter fractures will have

had a conductivity increase of up to 1000 mD. The influence of gel shrinkage on fracture conductivity is highly dependent on the fracture aperture. In wide fractures, severe conductivity increase may be seen even at low degrees of gel shrinkage.

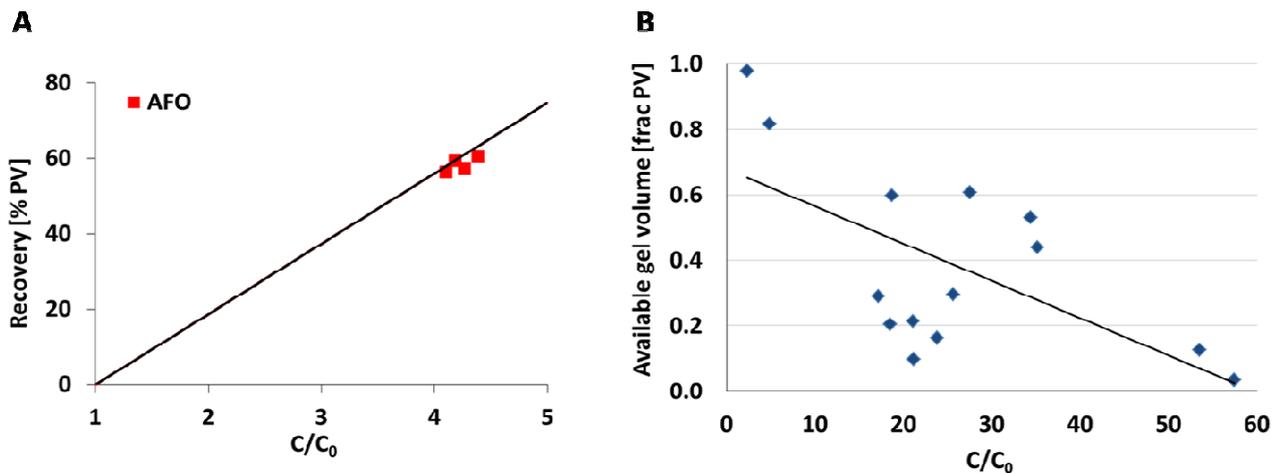


Figure 14: A) Concentration of the dehydrated gel layer after ended AFO imbibition was classified by weight measurements and volume recordings. B) Final concentration divided by original polymer concentration as a function of available gel volume for imbibition.

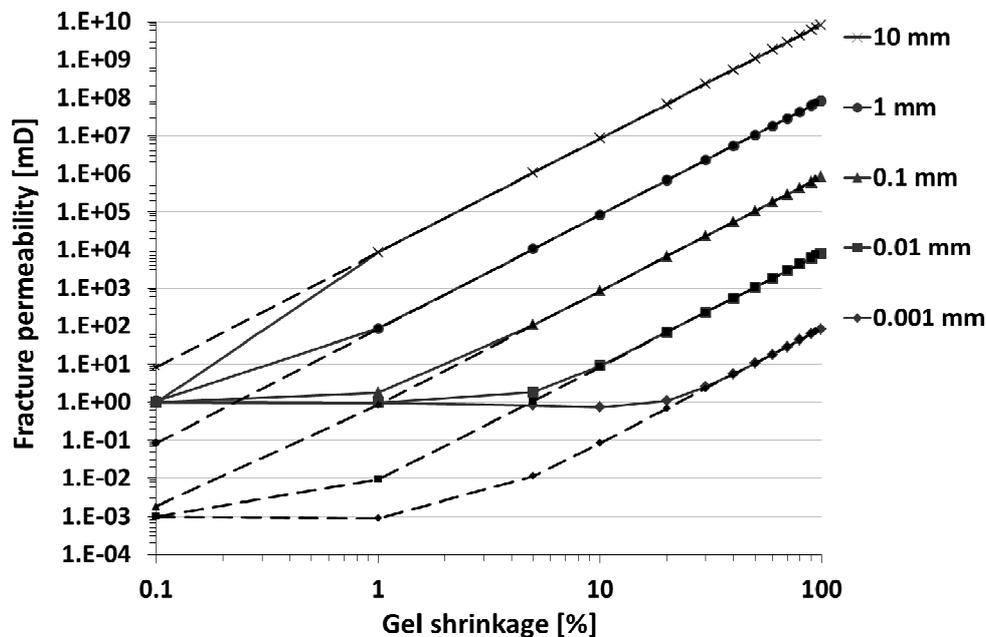


Figure 15: Fracture permeability as a function of gel shrinkage. The dotted lines reflect an initial polymer concentration of 5 %, which may be the case after gel placement with leakoff. The black lines are based on an initial polymer concentration of 0.5 %, which is also the initial value in all experiments.

Discussion

Imbibition of brine from gel was observed in all core materials and using both boundary conditions; AFO and TEO-OW. The results prove that the presence of capillary forces is sufficient to dehydrate aged polymer gel. This indicates that gel behavior during and after placement may be affected by properties that influence capillary forces; such as pore size, wettability and interfacial tension. It is therefore important to consider such properties to quantify and predict gel behavior in a reservoir.

Spontaneous Imbibition from Gel

The measured spontaneous imbibition rates suggest a difference between a gel/oil system and a conventional water/oil system. The differences were more pronounced in cores of higher bulk volume to open surface ratios (VSR), and included lowering of the initial imbibition rate with a factor of ten when the cores were submerged in gel. The decrease in recovery rate as a function of VSR was observed throughout all experiments. Changes in imbibition and production rate between water/oil and gel/oil cases are due to polymer gel characteristics or polymer gel/core surface interactions. The cross-linked polymer chains constituting the gel will not enter porous rock with the given pore throat radii, hence, water alone enter the cores by

spontaneous imbibition. The *in-situ* displacement process is thus regular two-phase water/oil flow. However, the imbibition of water from gel dehydrates the gel, building up a more concentrated gel film at the core surface. Seright (2006) defined a relationship between polymer concentration and filter cake permeability (Eq. 3), which states that increasing polymer concentrations decreases gel permeability. The decrease in gel permeability increases the skin factor on the core surface and induces an additional pressure drop across the concentrated gel layer, between the core surface and the fresh polymer gel. The end point for spontaneous imbibition, defined by zero capillary pressure on the capillary pressure curve in water/oil systems, may not be reached if the skin factor is high enough to counteract the capillary forces. At high VSR, for example in experiments utilizing TEO-OW boundary conditions, the skin factor will increase quickly. This may partially explain why the end point for spontaneous imbibition is not reached in some TEO-OW experiments by continued imbibition in gel. The dependency on the bulk volume available for imbibition compared to the open surface indicated that a faster imbibition process and a higher degree of oil recovery and gel shrinkage may be seen in highly fractured reservoirs compared to reservoirs of less fracture intensity.

Gel Shrinkage Caused by Spontaneous Imbibition

In gel treatments of some reservoirs, gel is injected over a relatively short period (for example, about 1 day for many production-well treatments) to reduce fracture conductivity. Several authors have reported gel treatments to lose efficiency over time (White *et al.*, 1973, Seright, 2003, Portwood, 2005). Our work clearly shows that the capillary forces acting in the matrix of oil saturated porous rock are sufficiently strong to dehydrate gel and cause it to shrink. The shrinkage of gel may open large parts of the gel treated fractures to flow and decrease the efficiency of the gel treatment over time. The reduced imbibition rates in gel/oil systems compared to brine/oil systems corroborate that the effects of shrinkage by spontaneous imbibition may be a slow process, and that it may require time before the effects of shrinking is manifested by reduced blocking efficiency.

In other cases, especially injection wells in naturally fractured reservoirs, gel injection occurs over the course of 1 to 4 weeks (Hild and Wackowski, 1999). For these cases, dehydration of the gel by imbibition may occur largely at the same time as dehydration during the gel-extrusion process. Consequently, effects of subsequent gel shrinking may be less important.

Implications of Imbibition to Gel Treatments in Fields

The results presented in this paper all feature cores with large volumes of gel available for imbibition, effectively constituting a large (infinite) fracture volume compared to matrix pore volume. In naturally fractured reservoirs the fracture volume is usually less than 3% of the total PV and frequently less than 1% PV. This implies that a small fraction of spontaneous imbibition from the gel filled fracture to the matrix may shrink the gel enough to reduce its blocking efficiency. The high VSR present in a reservoir also implies that the spontaneous imbibition process may continue for a long time, in fact as long as a positive capillary pressure, or a capillary pressure above the osmotic gel pressure, is present in the matrix. The list below defines cases of special concern, where the findings in this study may be crucial to gel treatment efficiency:

- 1) Oil bearing zones. The capillary forces are strongly dependent on saturation: in water-wet systems, areas of high oil saturation are most prone to imbibition. It may thus be expected that imbibition effects influencing gel treatments are most prominent in areas of high continued oil saturation: oil production zones, oil production wells etc. This observation may also be exploited in special circumstances: "good fractures" that allow water to be distributed deep into a reservoir may have high oil saturations in the adjacent matrix. So, imbibition that compromises the gel may promote water entry into and oil displacement from these areas during gel placement. In contrast, for areas with fractures that have allowed severe water channeling, high water saturations in the surrounding matrix may cause a lower level of gel dehydration, and therefore, the gel may still provide a substantial reduction in fracture conductivity.
- 2) Gel treatments using immature gels (gelant). When injecting gelant, the polymer and cross-linker concentrations will at best remain constant, but may also be diluted by formation water, experience cross-linker diffusion to the matrix etc. This means that the gel forming in the fracture has a concentration that is equal to, or lower, than the injected concentration. This study shows that the chances of imbibition occurring at typical gelant compositions are high and the gel treatment is likely to lose efficiency over time, providing a potential for spontaneous imbibition is present. Using formed gels, a leakoff effect may take place during extrusion through fractures, which increase the concentration of the gel. A higher gel concentration means a more rigid gel, where molecular bonds will be stronger. A critical gel concentration for this particular gel was observed to be $C/C_0 \approx 60$. At this concentration, an osmotic pressure in the gel is suspected to counteract further dehydration. Studies have shown that this concentration may be achieved during extrusion of formed gel through fractures, and a gel treatment using mature gels may thus be less prone to spontaneous imbibition.
- 3) Large fractures. Calculations performed in this work showed that gel dehydration will have a more severe effect in large fractures, and will be a concern for gel treatments using both immature and mature gels: the leakoff process during injection of mature gels is less efficient in larger fractures (fractures of higher aperture), and the gel concentration after placement thus lower. In gels of lower concentration, spontaneous imbibition effects are more likely to occur.

Adding surfactants to the polymer gel may decrease spontaneous imbibition of water by lowering the interfacial tension (IFT) between brine and oil. Surface active agents may also aid leakoff water to overcome the capillary threshold pressure imbedded in an oil-wet reservoir and improve dehydration of gel during extrusion through oil-wet fractures. Oil-based gels may also prevent shrinkage in regions where spontaneous imbibition of water is an important mechanism.

Conclusions

- Capillary forces in outcrop chalk, limestone and sandstone were strong enough to dehydrate aged polymer gel.
- A significant reduction in gel volume was seen due to spontaneous imbibition.
- The degree and rate of gel shrinkage were dependent on the available volume of gel present close to the core surface. When a gel volume less than the imbibition potential was available, gel shrinkage of ~ 98 % was recorded.
- The fastest gel shrinkage is experienced in the core with the lowest available gel volume for imbibition.
- Gel shrinkage may severely affect the gel blockage efficiency by opening parts of the fracture volume to flow. The influence of gel shrinkage on fracture conductivity is highly dependent on the fracture aperture and wide fractures will experience severe conductivity increase, even at low degrees of shrinkage.
- Gel shrinkage may be most severe 1) in oil bearing zones, e.g. oil production wells, 2) during field treatments using gelant, and 3) in wide fractures. One suggestion to avoid large scale shrinkage is to add surfactant to the polymer gel, thereby lowering IFT.
- The rate of spontaneous imbibition of brine bound in aged polymer gel was quantified for strongly water-wet chalk cores using both AFO and TEO-OW boundary conditions.
- The imbibition process was highly dependent on the presence of a dehydrated gel layer on the core surface, which introduce a skin factor during imbibition.
- Measuring the rate of spontaneous imbibition using AFO boundary conditions could be challenging, as the oil that was displaced from the core was trapped within the gel, either on the core surface or in the core vicinity. Care must be taken not to remove any part of the gel layer during weighing, as this will accelerate the imbibition process.
- In AFO experiments, even though the rate of imbibition was decelerated when a dehydrated gel layer was present, the end point saturation was reached in all experiments.
- In two ends open (TEO-OW) experiments, the rates of recovery were reduced and dependent on the bulk volume/open surface ratio, VSR. This ratio was equal to the core length for this particular setup.
- The endpoint for spontaneous imbibition was not reached during horizontal TEO-OW experiments by continuous imbibition in gel, due to the dehydrated gel layer on the end face representing a positive skin factor. When the gel layer was removed, spontaneous imbibition continued.

Acknowledgements

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Nomenclature

S_o = Oil saturation

$m_{gf}(t)$ = gel film weight as a function of time

$\Delta m(t)$ = weight differential as a function of time

m_i = initial core weight

$m_{(c+gf)}(t)$ = weight of core including gel film as a function of time

Δm_{max} = maximum weight change

$m_{(c+gf),max}$ = end point core weight, including gel film

$m_{gf,max}$ = gel film weight at the end of spontaneous imbibition

VSR = bulk volume to open surface ratio

V_b = bulk volume

A = surface area open to flow

PV = pore volume

IFT = interfacial tension

C_o = original, "injected" gel concentration

C = gel concentration after dehydration

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More pictures of first AFO cores during imbibition may be seen here: <http://shadayim.deviantart.com/gallery/8482304>