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## **Abstract**

Utilization of Steam Assisted Gravity Drainage (SAGD) features is currently one of the most widely applied technologies for production of bitumen oil in Canadian oil sand fields. However, as in most oil and gas reservoirs, the bitumen oil reservoirs also differ depending on their reservoir structure; for the Surmont field case in Athabasca oil sands region, the lack of natural sealing and presence of overlying thief zones became a point of concern for a conventional application of the SAGD method.

The conventional operational conditions of the SAGD process were considered to be less efficient or not applicable in the presence of the overlying thief zones and lack of natural barrier because of the potential energy losses from the steam chamber. There is also a high risk of the SAGD well pair shut-down due to influx of undesired fluids owing to pressure communication between the bitumen reservoir and the overlying gas and water zones.

This work is done as a part of an ongoing research seeking to resolve the thief zone issue, and aims to (a) develop the main requirements for chemical isolation method for the Surmont field and (b) evaluate the wide range of possible isolation products in order to find the appropriate systems satisfying the established requirements.

As a result of a literature review and communications with relevant technical experts, a preliminary screening was conducted by ranking the products based on their potential and possible technical restrictions for thief zone treatment. At this stage, products considered to having serious limitations for such treatment were eliminated from further evaluation. For example, cement was considered to having serious injectivity issues as result of particles size; simple inorganic salts not having enough plugging ability; and foam systems due to lack of yield strength. The few promising products (polymers, silicate, and epoxy resins) were chosen for a further experimental screening.

Based on the criteria identified for an effective treatment of thief zones, relevant experimental tests assisted to narrow down the possible isolation products by eliminating the aqueous gel systems (silicates and polymers) due to their instability at high temperatures. The experimental results indicated that the epoxy system is the most effective plugging agent. The laboratory findings also showed that the initially perceived epoxy system injectivity limitations can be resolved by a proper formulation of the epoxy components and relevant solvents.

# **1 Introduction**

## **1.1 Background**

Surmont is a heavy oil field located in northeast Alberta with an estimated oil in place of approximately 20 billion barrels of bitumen (Byerley et al., 2009). Bitumen production is extracted via a Steam Assisted Gravity Drainage (SAGD) technology which involves a continuous steam injection into the reservoir in order to liquefy and mobilize the bitumen oil, which can then be produced to the surface. In addition to the oil sands resource development, gas is also produced from shallow reservoirs located on top of the bitumen reservoir and separated by an aquifer. The gas and bitumen production have the potential for mutual interference.

The initial evaluation work regarding the use of the SAGD process at Surmont was performed by the Oil Sands and Research Division of the Alberta Department of Energy (AEUB, 2003). The study recognized that SAGD could not be directly applicable to Surmont and that a research and development program was required to advance the SAGD technology for a suitable application to extract the bitumen. Although some features of the SAGD process have been patented, many unique questions remain to be answered in order to develop this concept for the Surmont field. For this reason, the study recommended that an experimental pilot be developed for Surmont to address the areas of uncertainty.

Steam thief zones, defined as permeable zones that have high gas or water content and that occur in and/or adjacent to the bitumen reservoir, have a negative impact on SAGD operations because of the possibility of short circuiting the pressure in the steam chamber and because of the associated heat losses.

In 1997 a pilot project was initiated at Surmont to assess the feasibility of the SAGD recovery method for commercial development and evaluation of thief zones impact. This pilot facility utilizes three SAGD well pairs which are still producing today (ConocoPhillips, 2008).

## **1.2 Scope of study**

As part of ongoing research on thief zone assessment in Canadian bitumen oil reservoirs, the aim of this thesis work is to propose and justify possible products applicable for thief zone treatments in the Surmont field.

The first part of this work consist of scientific and patent literature review concerning the injection of products for the purpose of in-depth plugging of an reservoir sections with special attention to the thermal stability and isolation mechanisms. Following this review, laboratory procedures developed and used to assess the efficiency of zonal selected isolation technologies.

## 2. The Surmont field

### 2.1 General information

The Surmont field is located approximately 60 kilometers southeast of Fort McMurray, Alberta, in the Athabasca oil sands region (Fig. 2.1). Surmont is operated by ConocoPhillips Canada and is a 50/50 joint-venture with Total E&P Canada.

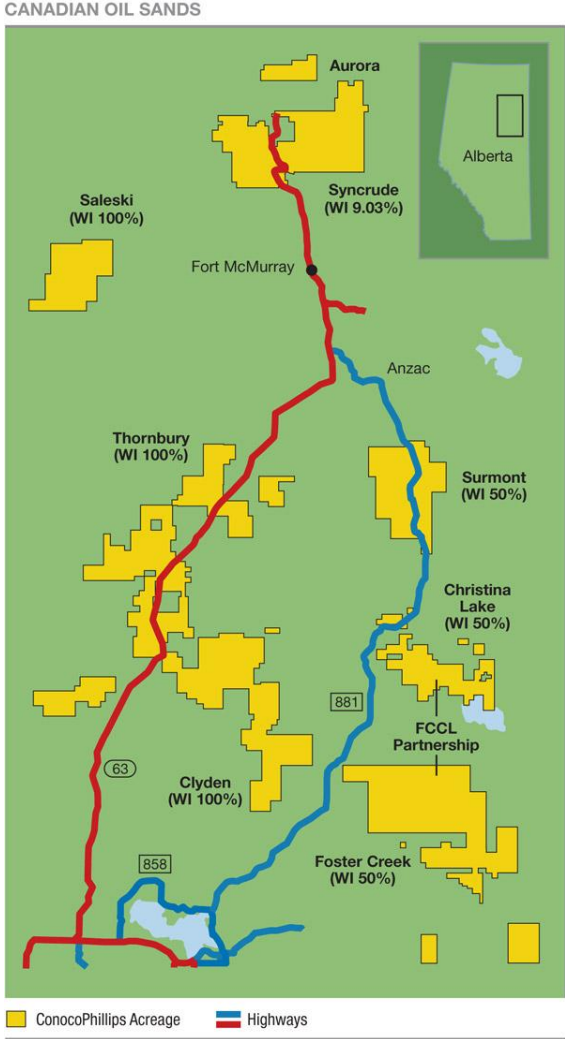


Fig. 2.1 – Bitumen oil fields in Canada operated by ConocoPhillips (ConocoPhillips Fact Book, 2012).

The oil sands formation is between 300 and 400 meters below the surface, with thickness varying up to 60 meters. The In-place bitumen is estimated at 20 billion barrels, with a potential recovery of 25 to 50 percent (NEB, 2004).

Surmont is being developed through a multi-phased approach. Phase 1 has a design capacity of 27,000 barrels of bitumen per day with a design steam-oil ratio of 2.25. It entered into commercial production in the 4th quarter of 2007 (Total and Conoco Phillips, 2010).

Surmont Phase 2 development is expected to take the total bitumen production to approximately 136,000 barrels of bitumen per day. Phase 2 was sanctioned in January 2010 with a production start-up currently scheduled for 2015. Phase 2 is currently under development (Total and ConocoPhillips, 2010).

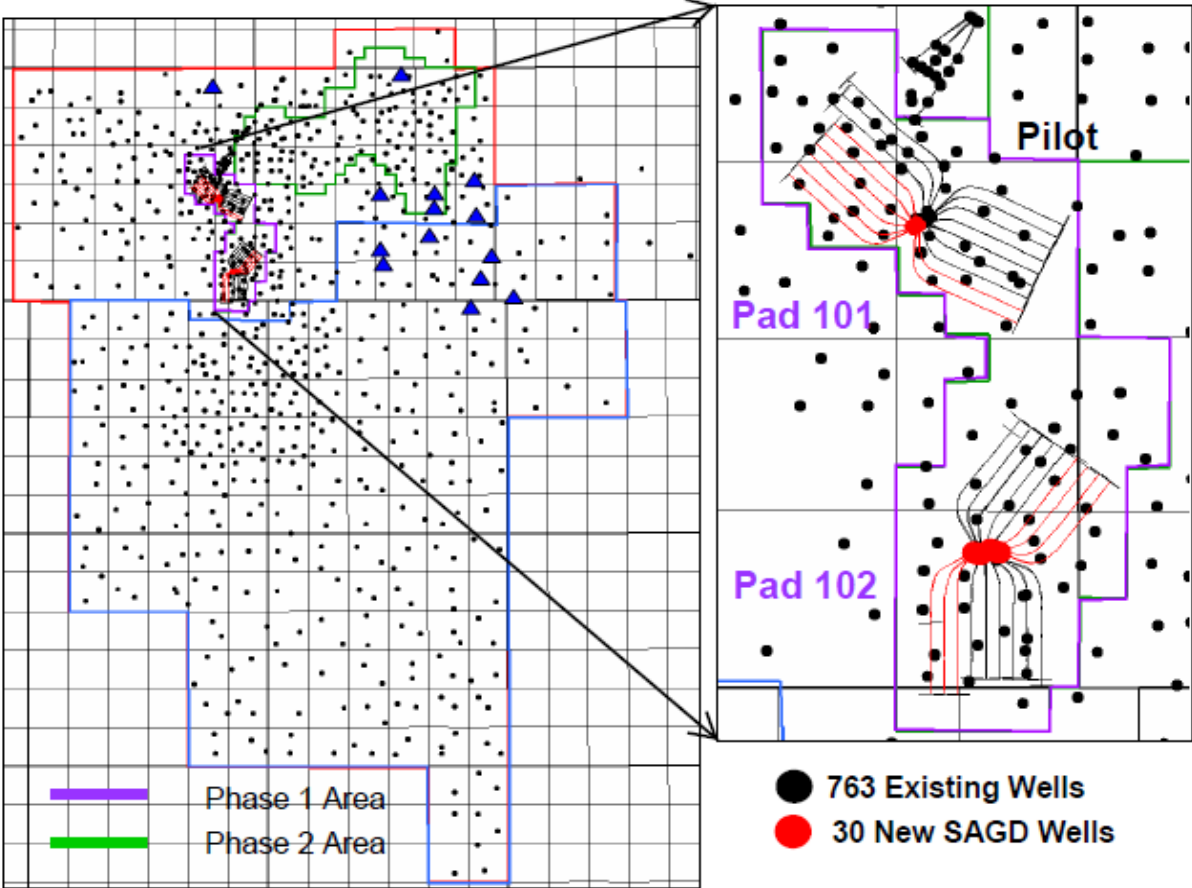


Fig. 2.2 - Well layout for Pilot project, Phase 1 and 2 (Surmont Oil Sands, 2009).

The Surmont leases consist of approximately 80 square kilometers and utilize SAGD for bitumen oil recovery from the McMurray Formation (Fig. 2.2). The production operations for each phase also feature associated steam cogeneration, well pads, water treatment and other related facilities.

## 2.2 Reservoir description

The McMurray reservoir is considered to be Albian-Aptian in age and was deposited during the Lower Cretaceous period. It consists primarily of unconsolidated sands between 30 to 70m thick in the Surmont area (Fig. 2.3). The porosity in the sandy units typically ranges from 30 to 35% with permeability greater than 1 Darcy. The bitumen typically averages 8° to 10° API. At in-situ conditions viscosity is in the 1 to 5 million cP (centipoises) range (Byerley et al., 2009).

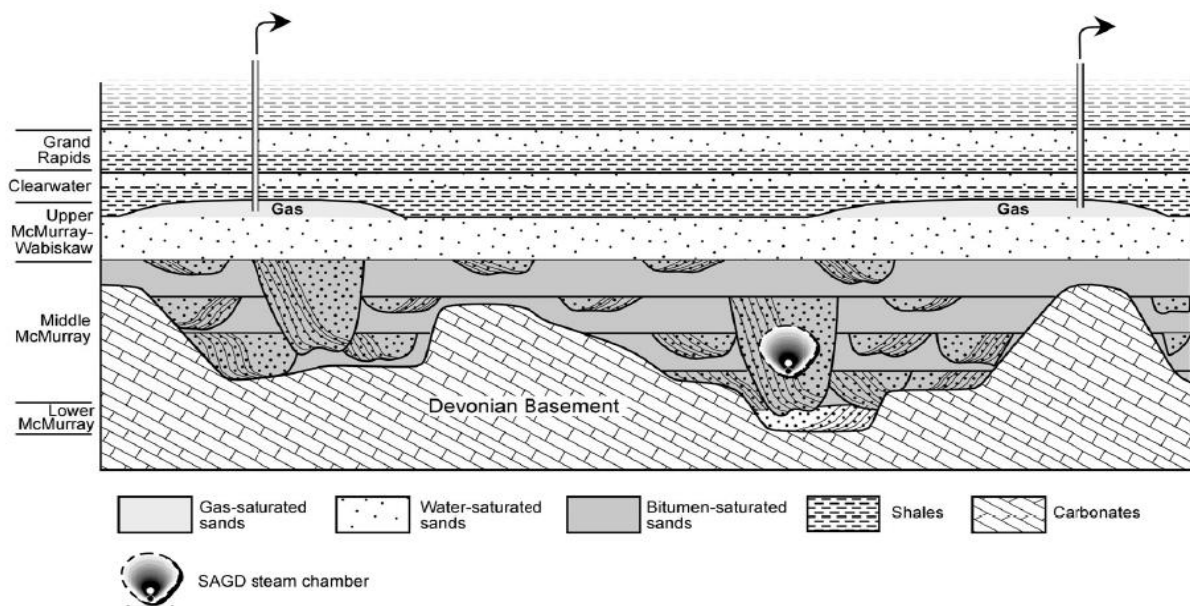


Fig. 2.3 - Diagrammatic representation of the geology, depositional characteristics, hydrogeology, and gas and bitumen production (Bachu and Haug, 2002).

Down cutting of the eroded Paleozoic surface during middle Jurassic to early Cretaceous created a large north northwestward trending regional valley system with associated tributaries, which was backfilled with transgressive estuarine deposits (Keith et al, 1998), forming the laterally discontinuous Lower McMurray. Stacked, prograding, shoreface parasequence sets were deposited on top of the Lower McMurray, which were subsequently incised by lowstand channels that filled with fluvial estuarine sands, silts and muds (Ranger and Pemberton, 1997). This succession is informally called Middle McMurray.

Reservoir-quality sands include minor amounts of interbedded silts or muds, and shale-clast breccias. The Upper McMurray, ranging in thickness between 3 and 20 m (Hein et al., 2000), is comprised of an upward coarsening succession of interbedded sands, silts and muds that were deposited under more brackish-to-marine tidal flat/lagoonal and estuarine conditions.

The Wabiskaw Member of the Clearwater Formation disconformably overlies the McMurray Formation and is comprised of thin, coarsening-upward continuous marine sandstones. Bitumen occurs within the lower portion of the Middle McMurray sands, reaching saturations of up to 85%, with the highest oil saturations commonly at the base.

Water is present to various degrees of saturation in the Lower and Upper McMurray sands, and gas pools are found in the Upper McMurray and Wabiskaw sands. The gas pools vary in size, from several hundred meters to a few kilometers across, and are located at distances of mostly a few hundred meters, up to 3-5 km, from each other (Gulf Canada Resources, 1999; Petro-Canada Oil and Gas, 2001).

**2.3 Gas over bitumen**

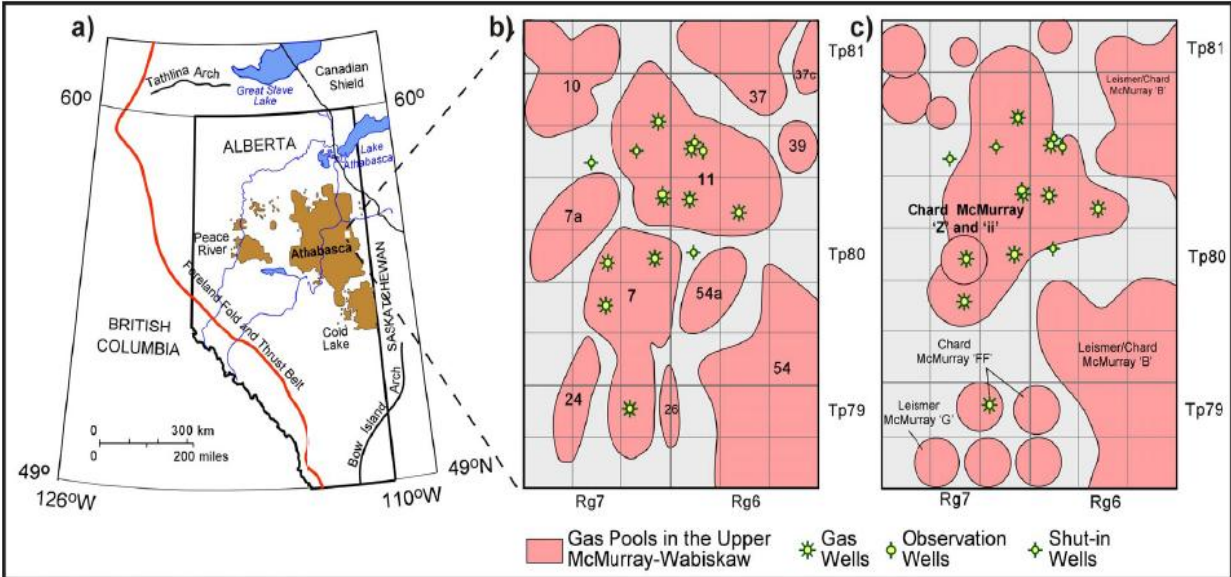


Fig. 2.4 - Gas pools overlying bitumen reservoir in Athabasca region (Bachu and Haug, 2002).

The Upper McMurray-Wabiskaw gas pools were originally in hydrodynamic equilibrium with the respective underlying aquifer and the regional boundary conditions imposed by the elevation of the recharge and discharge areas and the permeability distribution within the McMurray strata.

The initial relatively low pressure in the McMurray aquifers and gas pools already makes them potential steam thief zones if in contact with bitumen reservoirs (O'Rourke and Anderson, 1999).

Figures 2.5 and 2.6 show the occurrence of those zones based on log interpretations.

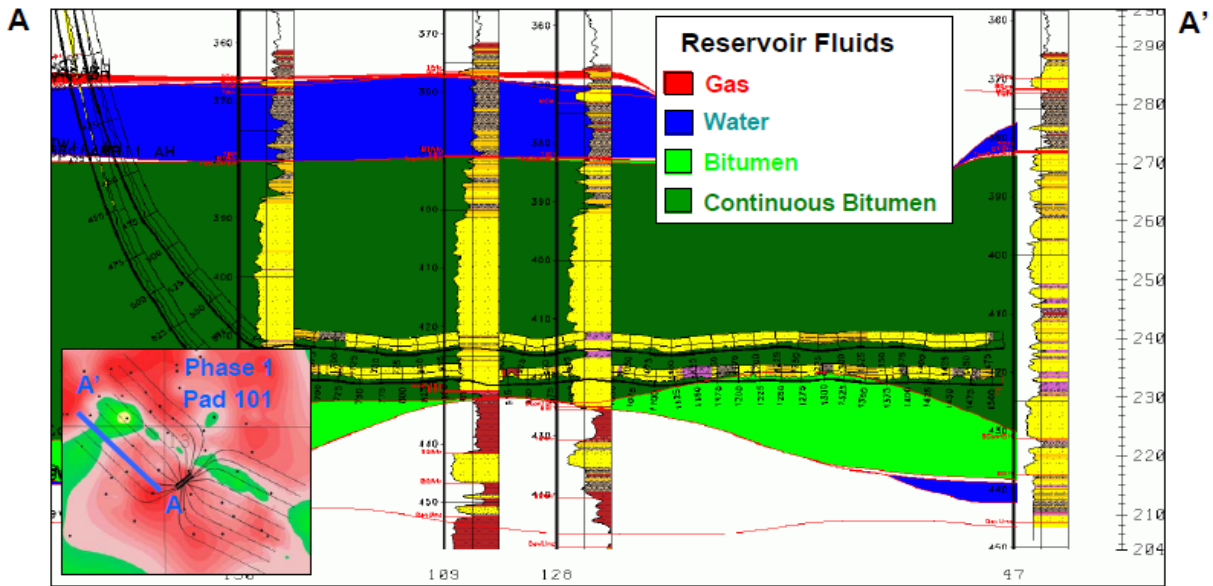


Fig. 2.5 - Example of overlying water bearing layer over bitumen reservoir (Surmont Oil Sands, 2009).

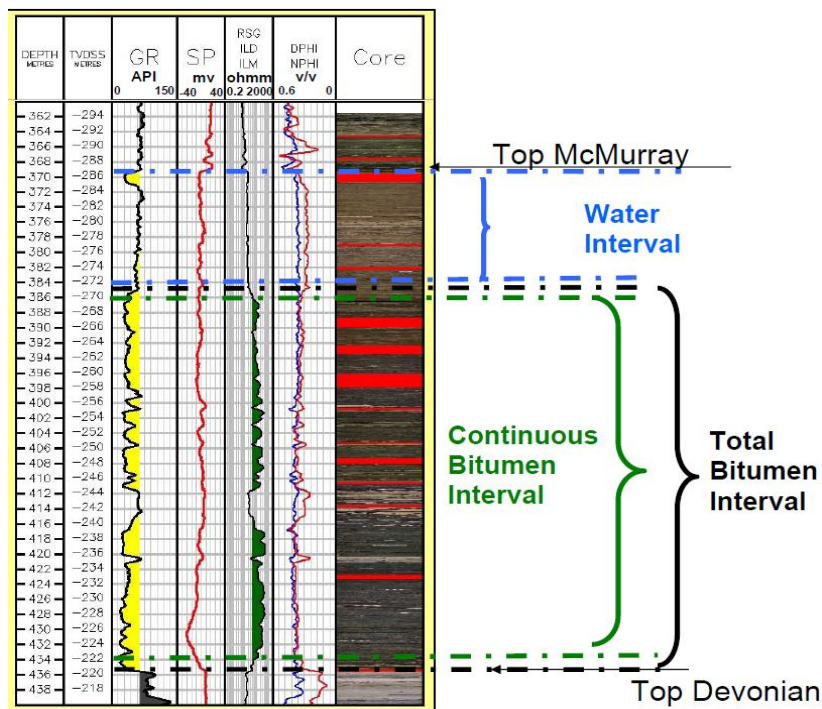


Fig. 2.6 - Example log of top water zone over continuous bitumen (Surmont Oil Sands, 2009).

If a steam chamber is unimpeded by reservoir heterogeneities such as shale layers, as in the case of stacked channel sands, it will expand all the way to the top of the bitumen-saturated zone. The high pressure steam will breach into the overlying water- or gas-saturated sands of the Upper McMurray-Wabiskaw aquifer, and the pressure in the steam chamber will drop to equalize with the pressure in these strata (Bachu and Haug, 2002).

As a result, the steam temperature will fall, with corresponding negative effects on bitumen viscosity. If the temperature drops below the bitumen-mobilization threshold (~60 °C) the SAGD operation will have effectively ceased, although it becomes inefficient and uneconomic.

The discussed thief zones range in volumetric size and occur in three different types:

- Gas over bitumen
- Water layer over bitumen
- Gas and water zones over bitumen

Figure 2.7 presents the net thickness of top gas and water layers in Surmont lease Phase 1.

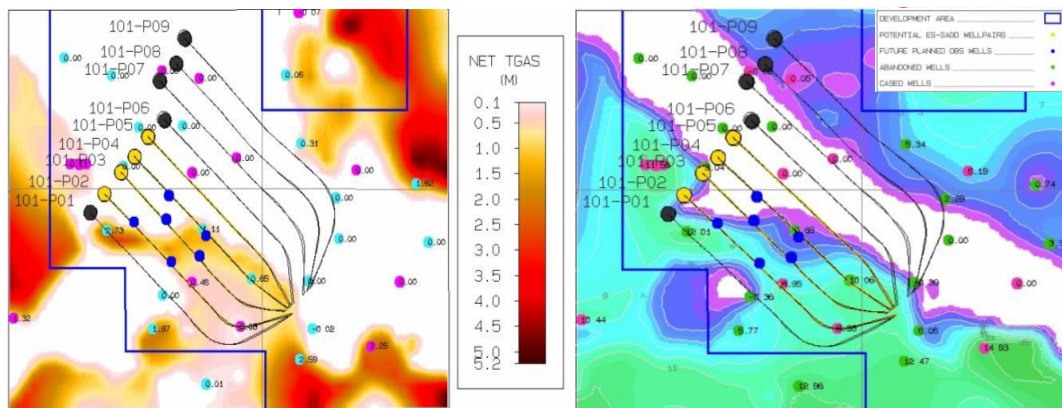


Fig. 2.7 - Top gas and water thickness in Phase 1 area (Surmont Oil Sands, 2009).

Schematic example of steam chamber interaction with thief zone and possible impact is illustrated in Figure 2.8. The worst case of such interaction predicts steam breakthrough in upper water and gas zones due to depleted pressure in these layers and following water migration in bitumen reservoir cooling the reservoir which could lead to SAGD pair shut-in.

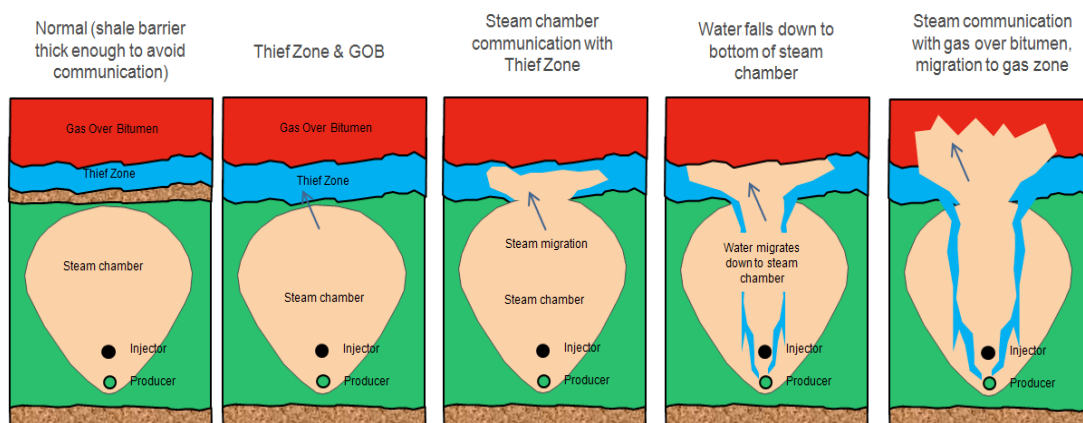


Fig. 2.8 - Thief zone interactions with SAGD operations (Thebault et al., 2011).



### 2.3.1 Top gas production

An additional important motivation for seeking the technical solution for thief zone problem lies in separated licensing for gas and oil producing operators. By Canadian regulations the bitumen oil and overlying gas in the same field could be produced by different companies with separate licenses.

This common practice became not applicable on fields with reservoirs where the direct pressure communication of gas zone and bitumen area could have negative interactions.

In 1996 the Alberta Energy and Utilities Board received submission from Gulf Canada Limited (now Conoco Philips) requesting the shut-in of associated gas production on its Surmont oil sand leases (AEUB and Gulf Canada Resources Ltd, 2000).

Following six years with additional geological studies and economic analysis the decision made to shut-in majority of the gas wells within a specified application area. The timeline of decision steps is shown in Figure 2.9.

The possible technical solution for the thief zones would allow gas production to resume in Surmont lease if the solution found could ensure no pressure communication and interactions between depleted gas zones and SAGD operations. With the current situation, such technical solution is vital requirement not only for future SAGD operations in areas with depleted gas zones, but would also allow unlocking the existing gas reservoirs within Surmont lease.



Fig. 2.9 - Gas over bitumen issue main steps and decisions timeline (Thebault et al., 2011).

### 2.4 Surmont Pilot project

The presence of potential thief zones in Athabasca oil sand reservoirs addressed the need for a pilot project in Surmont area. The Surmont project began as a pilot project with the construction of a small SAGD facility located near current operations. The pilot was built in 1997 and is still in use today to support existing operations and test new technology applications.

One of the main objectives of the pilot was to evaluate thief zone impact on SAGD operations by constant monitoring of steam chamber development and pressure development within the reservoir (Conoco Phillips Canada, 2008).

The time lapse 4D seismic is used to monitor development of the steam effects around the injection wells (Fig. 2.10). Detailed calibration of the 4D seismic anomalies to observation well data indicate that the seismic is responding to the softer lower viscosity heated bitumen that resides ahead of the steam chamber (Byerley et al., 2009).

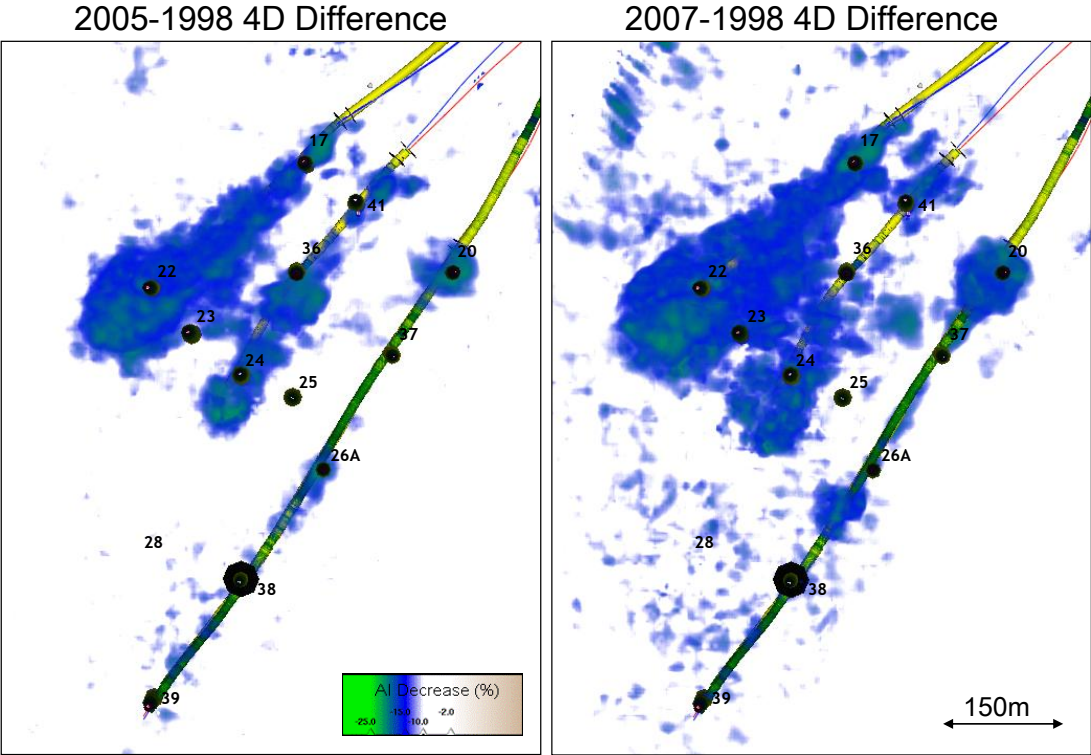


Fig. 2.10 - 4D Difference of steam chamber development for wells A, B, and C (Byerley et al., 2009).

High level of reservoir heterogeneity results in non-homogeneous steam chamber development within bitumen reservoir. The development of steam chamber is mainly controlled by decreasing/increasing operational pressures.

Example of potential steam breakthrough observed on 4D seismic picture of well C, Figure 2.11.

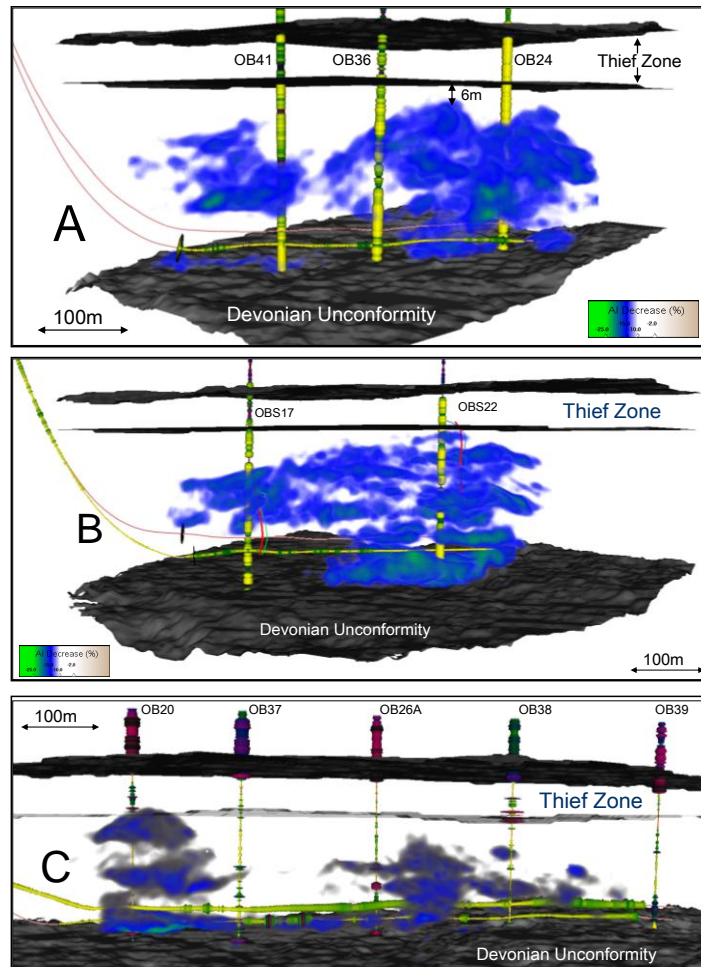


Fig. 2.11 - 4D Difference of steam chamber development for wells A, B, and C (Byerley et al., 2009).

One of the main outcomes from Pilot project is an evidence of thief zone direct pressure communication with the underlying reservoir. Even though the project proved possibility for SAGD operation under low pressure conditions the active steam chamber has not yet reached the thief zone, so the full impact of the thief zones cannot be determined (ConocoPhillips Canada, 2008).

## 2.5 Chemical Isolation method

The problem of thief zones presence over bitumen reservoir is currently the main challenge for many operators in the Athabasca region. One of the main control methods as reducing operational (steam injection) pressure would become irrelevant for reservoirs with more depleted gas layers over the bitumen due to low oil recovery and increased risk for negative interaction of steam chamber and thief zones (Planckaert, 2012).

As part of the ongoing research for the thief zone treatment, a new approach is considered in this work. The possible solution aims to utilize isolation methods to seal the bitumen reservoir area from overlying thief zones by a wide, lateral placement of isolation agents at the bottom of the thief zones (Fig. 2.12).

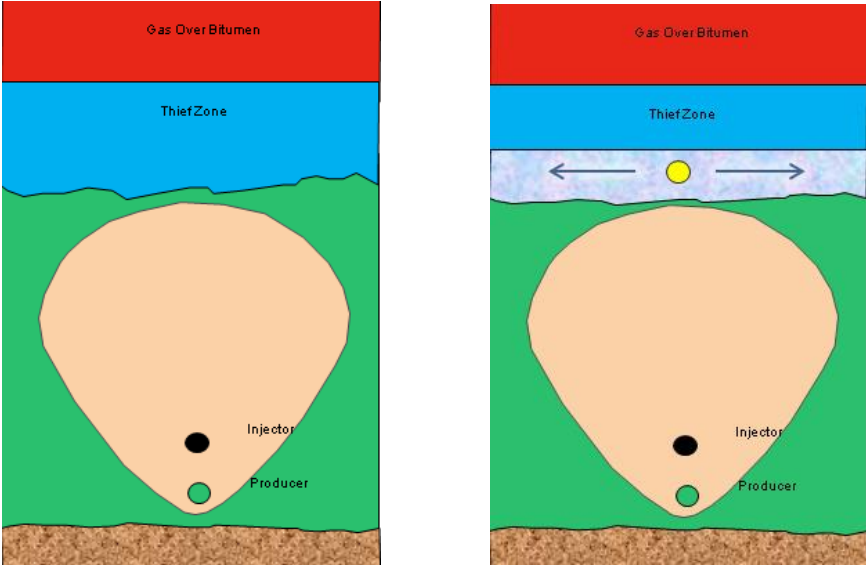


Fig. 2.12 – Schematic plan for isolation product placement at the bottom of thief zone (modified from Thebault et al., 2011).

If such treatment could be placed preventively it would work as conventional sealing cap rock, which would ensure no pressure communication between steam chamber in bitumen reservoir and overlying water/gas layers. The life-time of such sealing treatment should be practically as long as a SAGD well life-time (10-15 years), so that the isolation should have a good thermal stability during long period of time.

The main criteria for the placement of such agent would be:

- High injectivity (water-like viscosity and small particle size)
- High thief zone permeability reduction
- Thermal stability to exposed temperatures of 200-250 °C upon the arrival of the steam chamber.

The placement of such blocking agent initially assumed to be over maximum lateral area above continuous bitumen by drilling an additional horizontal well. Depending on the thickness of thief zone the volumetric calculation could vary significantly. The example

shown in Figure 2.13 is based on the average size of the SAGD pair drainage area would require the placement of chemical volumes somewhere between 1 to 2 million barrels.

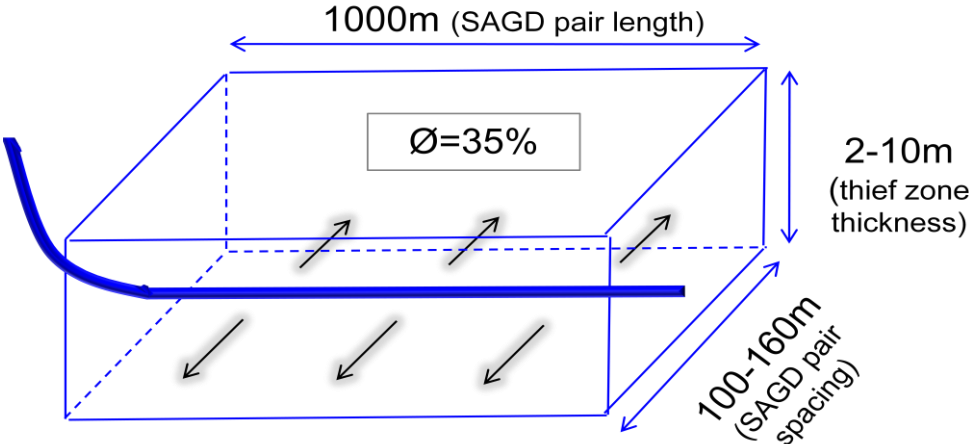


Fig. 2.13 - Wide lateral placement by additional horizontal well in thief zone.

Another approach is targeted treatment in reservoir areas where steam breakthrough is likely to occur. The placement in such case could be designed to be injected by a pattern of vertical wells to effectively perform product placement. Such approach is illustrated in Figure 2.14 with an estimated placement volume in order of 25 to 50 thousand barrels.

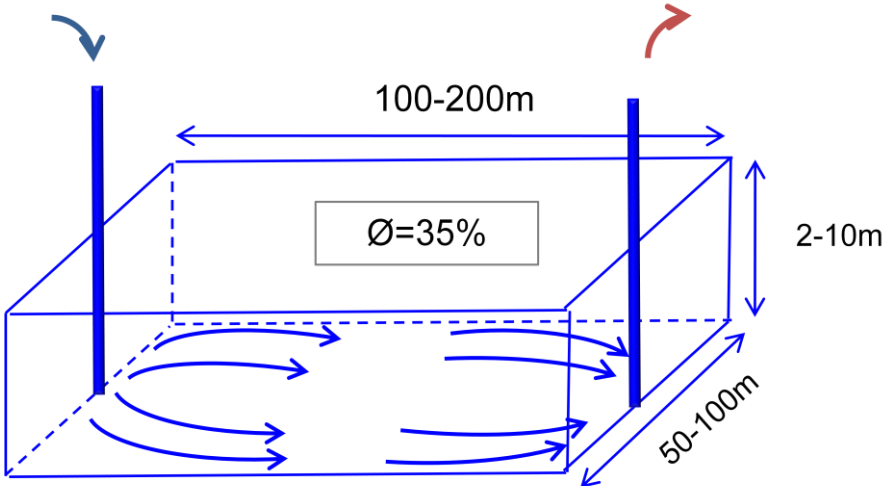


Fig. 2.14 - Placement volume in targeted treatment of thief zone.

The rough volumetric calculations could give a general estimation of the injection time and the economic requirements for the cost of the injected product. However specific task as injection time is not considered in detail in this work, but requirements for high injectivity and delayed isolation kinetics are taken into account on the initial screening stage.

### **3. Theory of conformance methods**

This chapter presents preliminary screening criteria developed based on information available in the literature by considering the effectiveness of the various systems according to the established requirements for the thief zone isolation and the SAGD field operational conditions.

Each conformance control method has a range of requirements/specifications to be met for each particular application. In this chapter a literature study is made for the methods which are considered to have a possible potential for thief zone isolation. Based on the screening criteria developed in this study, a preliminary evaluation could be performed to establish on whether the particular isolation method should be investigated further for the Surmont field or not.

#### **3.1 General theory on isolation agents**

Currently the oil and gas industry uses widely chemical treatments on almost all stages of petroleum operations. By nature all reservoirs are heterogeneous normally containing oil, gas and water; this results in reduced recovery of hydrocarbons, production of unwanted fluids, etc.

Reducing and controlling the production of unwanted fluids is a main concern in most of the oil and gas projects worldwide. A wide range of water and gas shut-off options is now available and are growing as technology evolves.

The main options for chemical isolation could be listed as follows (Kabir, 2001):

- Sealing Matrix and Small Fissures

Mechanical means like tubing/casing patch, straddle packer, and cement squeeze operations can provide a seal in the near-wellbore region. However, there are cases where it is desirable to achieve matrix or small fissure penetration of the sealing material. Examples include: small cement channels, natural fractures, and vertical coning through matrix. Cement squeezes cannot normally solve these problems.

- Selective Reduction of Unwanted Phase Flow

In the case of an offending high permeability zone with no crossflow in the reservoir, it is desirable to seal off the offending zone while allowing the oil zone to flow. In such case,

mechanical isolation or reperforation of the oil zone is required if a sealant (e.g., cement) is chosen. Alternatively, a relative permeability modifier (RPM) can be pumped which does not isolate offending zone but it reduces the water permeability much more than oil permeability.

- Subsequent Deeper Zone Perforating

Sometimes a depleted zone must be sealed off before adding perforations in a deeper zone. One option is to use a cement squeeze and then drill-out the hardened cement. However, this operation can be expensive and it may be possible to use a chemical to seal off the depleted zone. Then, in most of the cases, the chemical can be washed out from the wellbore quite easily and at a relatively lower cost.

- Reservoir Conformance

In case of reservoir flooding, sometimes mobility control or blockage of highly permeable flow paths is beneficial, which requires chemical means.

The main chemical options by functionally are as follows (Kabir, 2001):

1. Sealants (temporary or long lasting).
2. Relative permeability modifiers (liquid).
3. Weak sealant relative permeability modifiers.
4. Mobility control or flow diverting chemical flooding systems (viscous/ foam flooding, selective plugging).

Different types of sealing chemicals give a wide range of possibilities for choosing the appropriate and effective product for a specific field application and the systems which are recognized to be most flexible by formulation and operational properties became somewhat most popular within oil industry.

### **3.2 Conformance agents**

Understanding the mechanism and nature of each product is important to effectively use the available solutions for specific water and gas shut-off operations. Based on established criteria for potential isolation systems, the initial group of products is identified for further considerations. The preliminary review of each product with evaluation of effectiveness or

possible drawbacks is done based on material available in literature and personal communications with relevant experts.

### **3.2.1 Silicate gels**

Silicate gel is the oldest system used by industry to enhance water flood efficiency. The first mentioning of silicate gels as diverting agent is patented by Mills in 1922. Despite the continuous research and evaluation of silicates for water shut-off reported in recent scientific papers and new patents, there were only few field applications.

Krymrine and Boyce (1985) presented the chemistry of different silicate systems and summarized the findings of available papers and patents regarding on sodium silicate chemistry applied to oil field and grouting applications. The authors drew attention to a controversial fact that the silicates were inequitably neglected in commercial applications in favor of polymer treatments.

The field application of silicate with polymers in 1980 and 1998 in Hungarian projects on more than 100 well treatments was described by Lacatos who concluded that the applied methods were inexpensive, flexible and adaptable for wide range of production/injection problems.

Herring and Milloway (1999) reported successful gas shut-off applications by silicate gels in the Prudhoe Bay field of Alaska stating that large volumes of silicate gel provided an impermeable barrier against the gas influx at high temperature conditions.

Another field application of silicates for deep treatments was the ones on the Gullfaks field in North Sea described by of Lund et al. (1995).

#### Chemistry of silicates

Silicate systems are composed of high ratio liquid sodium silicates,  $\text{Na}_2\text{O} : n\text{SiO}_2$  ( $n > 3$ ), and one or more initiator chemicals which promote formation of a gel. Metal ions such as calcium, can cause immediate precipitation of water-insoluble metal silicate, which addresses the need of large preflush before silicate placement. Most of the initiators are acidic, water-soluble chemicals which lower the pH of silicate solution and promote liquid silicate gelation through polymerization of silicate species (Jurinak and Summers, 2001).



Other organic chemicals such as aldehydes, amides, and esters undergo hydrolysis in water to form carboxylic acids. Vinot et al. (1989) described the formation of silicate gels by hydrolysis of a diester for controlling the gelation kinetics.

The properties, chemistry and gelation of silicates are exhaustively discussed by Iler (1979). In his work he described silicates polymerization steps from monomers to large particles and gels as shown in Figure 3.1. Nano-size particles of silicates ensure good injectivity allowing deep placements with consequent plugging and permeability reduction by formation of in-situ sol or by plugging the pores by growing silica aggregates.

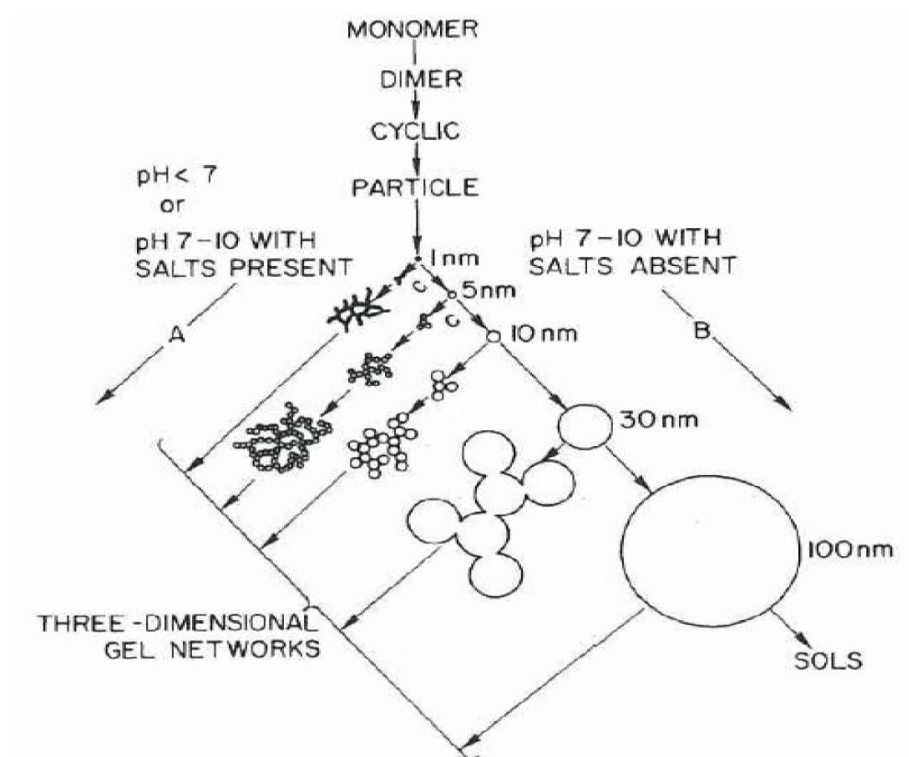


Fig. 3.1 - Polymerization behavior of silica (Iler, 1979).

The gelation of silica is believed to result from particle collision, bonding and aggregation into long chain networks. Particle collision is promoted by lowering surface charge (reducing the pH), by charge screening (addition of cations), increasing particle concentration or by increasing temperature.

### Gelation kinetics

As mentioned, the silicate gelation kinetics depends on pH variations, which on practice could be controlled simply by amount of acidic activator added into solution. Silicate gelation time

versus pH and salinity of solution is shown in Figure 3.2. It can be seen that even small variations in the pH have large impact on gelation time.

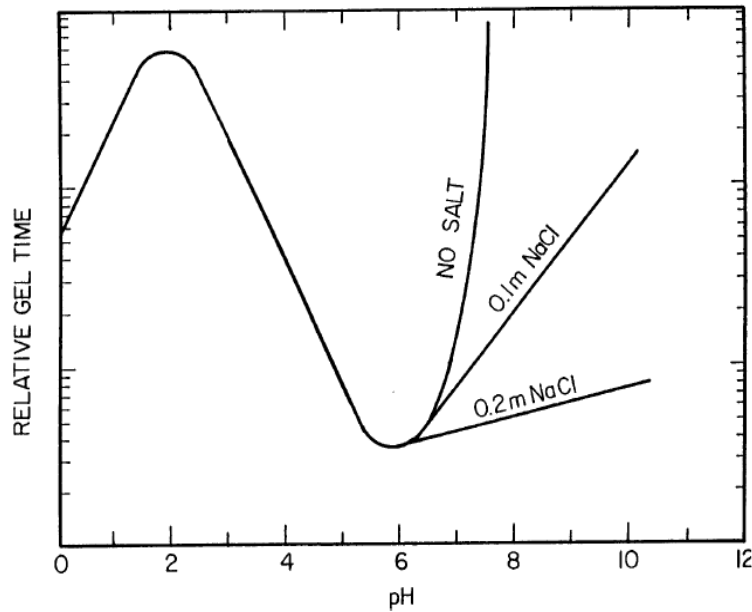


Fig. 3.2 - Relative gelation time vs. pH and salinity (Vinot et al., 1989).

The gelation time also sensitive to the brine salinity and it decreased with increased salinity of makeup water. Another important parameter affecting gelation is temperature, which could be also effective controlling tool for gelation kinetics.

Assuming bulk gelation kinetics and taking into account key parameters affecting gelation Stavland et al. (2011a) proposed the following form for gelation time:

$$t_{gel} = A \times e^{\alpha[Si]} \times e^{\beta[HCl]} \times e^{\gamma\sqrt{[Ca^{2+}]}} \times e^{Ea/RT} \quad (3.1)$$

where:  $t_{gel}$ = gelation time [days]

[Si]= silicate concentration [wt%]

[HCl]= HCl concentration [2M wt%]

[Ca<sub>2+</sub>]= concentration of calcium [ppm]

T= absolute temperature [K]

Considering silicate injection and gelation in reservoir conditions some of the key parameters should be taken into account.

Designed concentration of silicate and activator may be altered during deep placement due to dispersion by mixing occurring between displacing and displaced phase, which practically could be solved by careful design of injected concentrations. Also the buffering capacity of reservoir rock may affect the planned pH reduction of silicate solution, which will be important factor for estimation the expected gelation time of silicates.

In coreflood experiments on silicates reported by Stavland et al. (2011a) reported longer gelation time in sand packs ( $k=10D$ ), which he explained by partly retention and partly high permeability.

Another finding by Stavland et al. (2011b) for flood experiments is increased rate of deposition of silicate particles by decreasing the injection velocity. This observation may be relevant to consider for deep placement when the injected silicate solution will reach a depth where the velocity is significantly reduced.

#### Gel properties and stability

Once gelation process finished silicate solution forms strong ringing gel. The yield strength of gel generally depends on silica and activator concentrations. Even small concentrations of silicate were able to form a strong enough gel. The 4 wt% concentration gels reported to withstand pressure gradient of 10 bars per meter in experiments with sandpicks (Stavland et al., 2011a).

One of the disadvantages of silicate gels is the brittleness of formed gel, which makes the gels ineffective for blocking void spaces and fractures, but more competent and strong in matrix-filling applications.

Another drawback of silicate gels is shrinkage and syneresis with time (water expulsion). This process may result in reduced permeability reduction for long-term applications (Vinot et al., 1989). The experimental work with silicate gels confirms water expulsion but in very small amounts, which should not affect seriously blocking ability of moderate size gel plugs (Stavland et al., 2011a).

Silicate gels are not susceptible to attack by mechanism that can cause degradation of many organic gels, such as bacterial action, hydrolysis and thermal decay. The only considerable weakness of silicate gels is solubility in high pH solutions (Jurinak and Summers, 1991), Fig. 3.3.

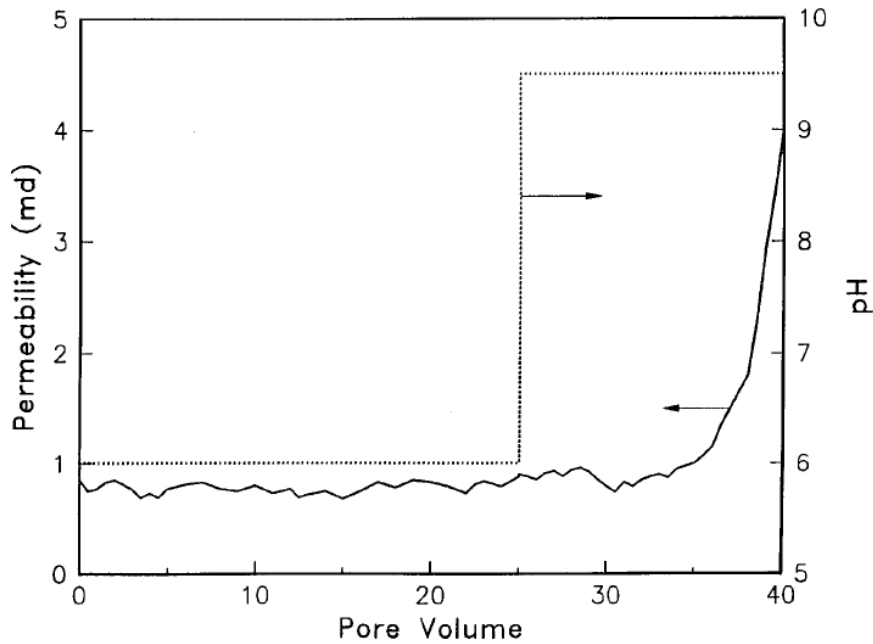


Fig. 3.3 - Gel permeability with high pH fluid injection (Jurinak and Summers, 1991).

Silicate gels are reported of having good thermal stability at high temperature conditions. Jurinak and Summers (1991) experimental sandpack results with silicate gel have shown a stable permeability reduction for temperatures up to 200 °C with constant flow of water through the gelled sandpack.

#### Silicate gel potential for thief zone isolation

The silicate systems considered to have high potential for thief zone shut-off application as providing high permeability reduction and also being inexpensive, environmentally friendly product. The water-like viscosity and no shear degradation allows deep placement of silicate solution in the treatment area.

The formulation of activator and effect of salinity and temperature on silicate gelation are important parameters for evaluation to ensure delayed and controlled gelation kinetics. The specific feature of SAGD process, the growing steam chamber with development of vertical thermal gradient in reservoir, may be also used for activating the gelation of a silicate solution.

The thermal stability of silicate gels upon steam chamber arrival is a critical parameter as the temperature of injected vapor is around 200-250 °C. The possibility of decreasing permeability reduction and steam shortcutting through gel layer at high temperature conditions should be verified by designing the gel system for field-scale applications.

### 3.2.2 Polymer gels

Polymers are macromolecules, made up of repeating units joined together. Molecular weight ranges up to several million Daltons. The physical size of the polymer molecules can range from sub-micron to a micron range (Dalrymple et al. 1999). Since their first applications in the 1970s, many organic polymer gel systems have been developed for water and gas shut-off operations.

Polymer systems extensively used in this last decade (Serigth, 1995; Sydansk and Southwell, 2000) , with polymer solutions injected into production and injection wells which after given time turn from low viscosity fluid to strong or weak gels depending on their formulations.

Amount of field applications with polymer systems is enormous, the variety of problems is solved with polymer systems range from injecting viscous solution with aim for increased sweep efficiency to diversion or total blocking of undesired phase flow.

Despite of the specific drawbacks, polymer solutions are recognized by the industry as very flexible systems whose efficiency is proved by hundreds of successful field treatments and experimental research.

#### Chemistry of polymer gels

Polymer gel systems start as a flowing mixture of two components - high molecular weight polymer and another chemical called a cross-linker. At some trigger, the crosslinking molecules start attaching themselves to two polymer molecules chemically linking them together. The result is a three dimensional network of interconnected polymer molecules (i.e. cross-linked) that stops behaving like a fluid and can eventually form a rigid, immobile gel. Figure 3.4 shows examples of monomer, 2-D, 3-D polymers and crosslinking visualization.

The most important polymer gel systems used in the oil industry can be classified as follows:

- Polyacrylamides
- Co-polymers
- Biopolymers

Cross-linking agents are of two types:

- Metallic ( $\text{Al}^{3+}$ ,  $\text{Cr}^{6+}$ ,  $\text{Cr}^{3+}$ , etc.)
- Organic

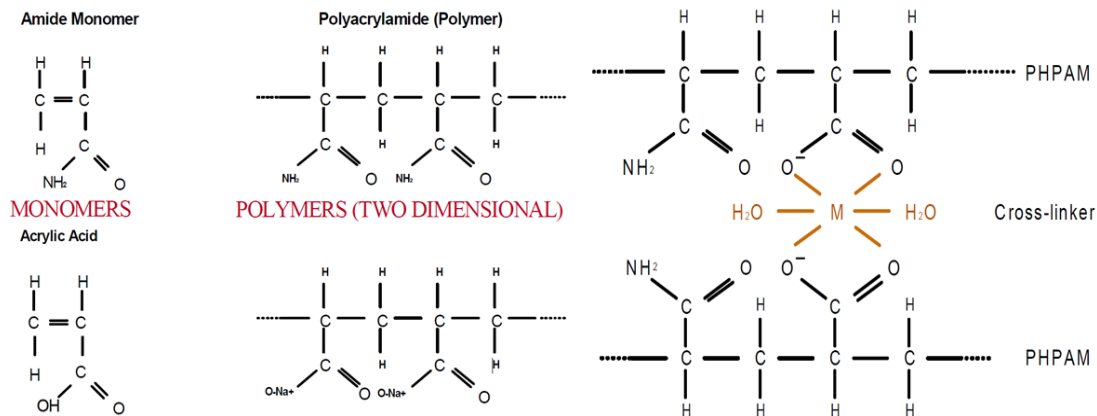


Fig. 3.4 - Monomer, Linear and 3D cross-linked polymer examples (Kabir, 2001).

### Gelation kinetics

Anionic polyacrylamides can be cross-linked with multivalent cations to produce gels. The rate of cross-linking and gel formation strongly depends on the charge density of the polymer as well as the source of tri- or tetravalent cations. Other factors affecting the rate include temperature, pH, salinity and the concentration of polymer and cross-linker.

Gel formation also shows strong dependence on the maturation time. This dependence indicates that crosslinking processes are very slow (Nijenhuis et al., 2003). Lockhart (1994) presented reaction of gel network formation in a HPAM/Cr(III) solution could last more than half a year. Other research by Hurd and Letteron (1932) indicates that gelation starts very slowly and when the gel is set, the process continues for a long time. Several methods for the determination of the gel point are presented in the literature (Nijenhuis, 1997).

### Gel properties and stability

Polymer systems have been most successful in near wellbore matrix and fracture treatments in low to moderately high temperature reservoirs. In fracture treatments, theory and many field results have shown that gel treatments are very effective in mitigating severe fluid channeling problem. However, polymer systems tend to undergo chemical modifications with time. One of these chemical instabilities is known as syneresis.

Syneresis causes shrinkage in gel volume, and consequently, water is expelled from gel structure. Depending on composition, degree of syneresis may be as little as 5% of the initial solution volume (Bryant et al. 1996). As the cross-linker bonds to reactive groups on polymer chains, the effective molecular weight of the polymer increases. Above some threshold, the solution becomes a viscoelastic solid. Where too much cross-linker is present, cross-linking

continues well past the point of gelation. This causes the polymer gel to contract in volume (synerese) expelling water.

Another reason for gel syneresis is polymer hydrolysis at high temperature and interaction with divalent ions. The polyacrylamide based polymers are exposed to hydrolysis at elevated temperature, with degree of hydrolysis depending on temperature. Hydrolysis converts acrylamide groups of the polymer backbone to the acrylate functionality, whose interaction with divalent cations can lead to syneresis of polymer gel. Therefore for polymer systems syneresis is a strong function of temperature and divalent ion concentration.

The factor which may affect final expected strength is a shear degradation of polymer systems. Shear degradation may occur as polymer passes through a pump, in turbulent flow through tubing and pipes, through perforation tunnels and formation pores. This fact addresses the requirement for controlling the maximum injection rate in field applications.

During the placement of polymers in porous media, the possible problems could occur due to adsorption in pore walls. Both the polymer and cross-linker have tendency to adsorption in porous media; if the adsorbability of two components are different, the ratio of polymer to cross-linker may change during the treatment. In such case the designed gelation time may be altered from expected kinetics.

#### Polymer gel potential for thief zone isolation

Numerous successful field applications of polymers as blocking agents and the variety of possible formulations of those systems make polymer gels the most attractive available solution to this date. Listed drawbacks are well understood in laboratory research and well managed in actual field applications.

The controlled gelation kinetics and blocking ability of polymer gels assumed to have potential for deep placement and blocking the thief zones. The delayed gelation is achievable by proper designing polymer type and crosslinking chemicals as well as taking into account residence conditions and temperature front which could be used as additional control tool for promoting the gelation before the arrival of the steam front.

Specific point of concern is the thermal stability of the polymer gels exposed to long-time contact with injected steam at elevated temperatures (200-250 °C). The effect of such high temperatures on the degree of hydrolysis and subsequent syneresis is to be verified by further evaluations of polymer application on Surmont field.

### 3.2.3 Resins (Epoxy)

Thermosetting resins are also used in water and gas shut-off applications, but not as commonly as the conventional gel systems. Resins provide much greater physical strength to seal fractures, channels and perforations compared to conventional gel systems and have been also applied in oil field, in less concentrated form, for sand consolidation (Bezemer and Meijjs, 1966).

#### Types of resin

Three most commonly used resins in field applications are the phenolic, epoxy and furfuryl alcohol resins.

Phenolic resins are thermosetting resins formulated, and partially reacted, before shipment to the field and they are stable at ambient temperature storage for up to two months. A catalyst, added before pumping, causes the liquid plastic to react at bottomhole temperature. The catalyst may be an acid or a base depending on the type of the phenolic resin used. Once phenolic resin has been fully reacted, it is strong and inert and only a strong caustic will attack it. If it is used where additional strength is required, such as to fill a channel or perforation, sand or silica flour may be added to the plastic before pumping. When this is done, another additive should also be added to aid the bonding between plastic and sand or silica flour. The thermal stability of phenolics after curing is reported to be about 400°F to 450°F (Kopf, 2003).

Furfuryl alcohol normally is not thought of as a polymerizable resin by itself, although there are many commercial furan resins based on similar chemistries. Furfuryl alcohol is used as a reactive solvent with phenolic and furan resins. This mixture polymerizes to a thermoset, but weak, brittle plastic in the presence of acid. Early attempts to use furfuryl alcohol for water shut-off met with difficulties, since its reaction with acid is very rapid. It is explosive at high temperatures and atmospheric pressures and is difficult to control in a well.

A controlled catalyst system (Sparlin and Hagen, 1984) using trichlorotoluene and pyridine with furfuryl alcohol allows safe, controlled shut-off treatments. Since furfuryl alcohol is readily available and relatively inexpensive, this system is a viable option for injecting a large quantity of material into a formation. Once it has reacted, the material is inert and has sufficient strength to resist movement of fluids through pores. It has higher thermal stability than phenolics or epoxies and has been reported to be stable in dry steam at 600 °F (Hess et al, 1971).



Epoxy is another type of thermosetting resin, commonly used as an adhesive and for potting electrical components. It has greater strength than phenolic or furfuryl alcohol resins, but is more expensive. Epoxy resin normally is diluted with a reactive solvent EGMBE known in the oil field as mutual solvent (Kabir, 2001)

The requirement for high physical strength plugs in water and gas shut-off is rather rare and not very common, hence very few papers existing on field application of resins as blocking systems. Knapp and Welbourn (1978) described acrylic/epoxy system for steam thief zone plugging. The developed systems claimed to have low viscosity with gelling time up to a month, which could form rubbery plastic like material and remain stable at elevated temperatures.

#### Chemistry of epoxy resins

Epoxy is a thermosetting polymer formed from reaction of epoxide and polyamine, more commonly called resin and hardener. Several types of hardeners utilized as activator by adding to a liquid epoxy resin before injecting. Most common epoxy resins are products of reaction between epichlorohydrin and bis-phenol A and common hardener is diethylenetriamine or triethylenetetramine. With mixing of these chemicals, the amine groups react with the epoxide groups to form covalent bond. Each NH group can react with epoxide group, which resulting polymer is heavily crosslinked forming rigid and strong material (Bhatnagar, 1996).

#### Epoxy hardening kinetics

The polymerization process also called “curing”, which can be controlled by temperature, resin and hardener type and their ratio in solution. Depending on these parameters hardening process can take minutes to hours or remain in liquid form and activated with high temperature.

#### Properties of epoxy

The epoxy can be cured with different curing agents depending on properties desired in the finished product and curing conditions. After hardening, the epoxy forms a high strength material which withstands tensile forces greater than 10000 psi. Cured epoxy resin is resistant to a broad range of chemicals, including caustic acids, fuels and solvents (Momentive, 2005).

### Viscosity of epoxy formulation

The epoxy resins are naturally highly viscous and solvents or diluents are used to lower the viscosity of epoxy resins systems. The solvents and diluents are low molecular weight liquids which are compatible with epoxy resins and curing agents (Petrie, 2005).

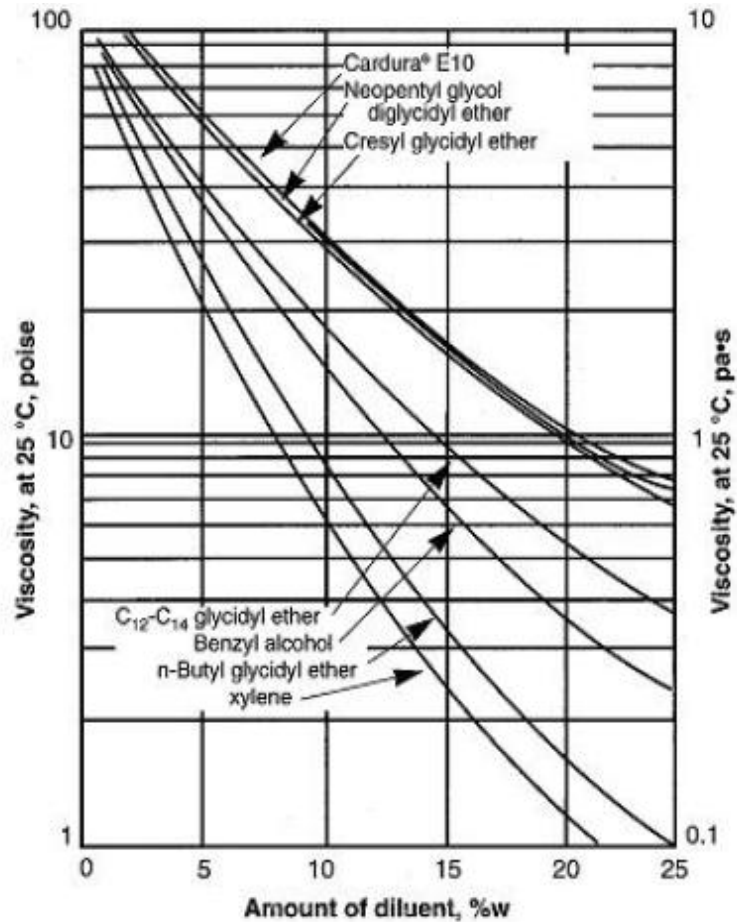


Fig. 3.5 - Viscosity at 25<sup>0</sup>C of EPON Resin 828 with various diluents (Momentive, 2005).

The viscosity of epoxy resins is decreasing with increasing temperature; the viscosity dependency on temperature is shown in Figure 3.6.

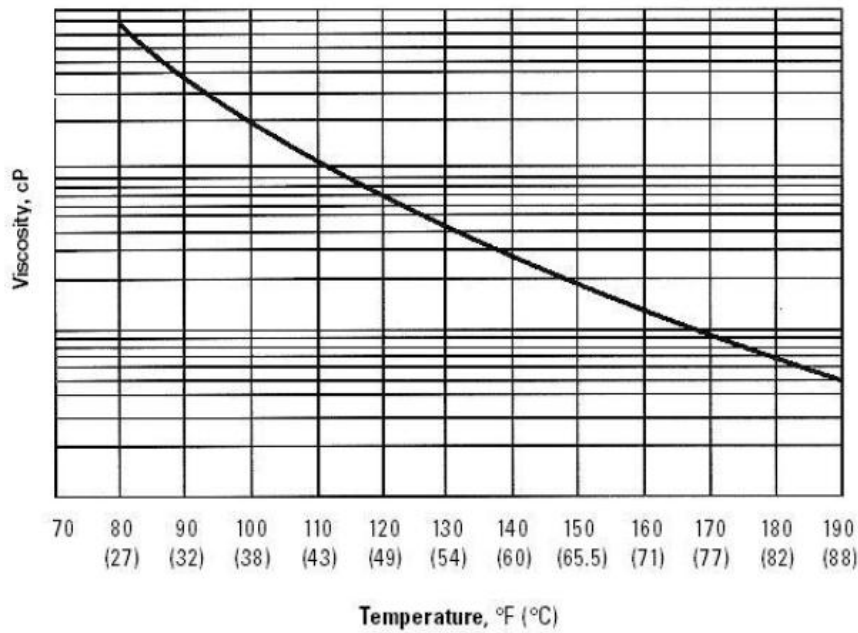


Fig. 3.6 - Viscosity – temperature profile for EPON Resin 828 (Momentive, 2005).

#### Epoxy resin potential for thief zone isolation

The hard and strong material forming after hardening the epoxy seems to be most reliable blocking systems for long time isolation of thief zone. Such isolation has possibility to withstand any induced pressure difference within SAGD process ensuring no steam breakthrough in overlying water and gas layers.

Another critical factor as thermal stability makes such resin isolation perfect candidate for such treatment, while other common conventional systems have some instability issues at elevated temperatures.

The thermosetting feature could be very effective controlling tool for delayed hardening. This allows preventive injection of liquid epoxy over the bitumen area, which will start hardening only when temperature start to rise due to in-situ thermal gradient and form impermeable hard material when the steam chamber reaches top bitumen level.

The possible restriction for epoxy application is its commonly high viscosity (11000 cP), but the solutions with lower viscosity are also available. Using solvents or emulsion of epoxy could be also practical method for solving the injectivity and placement issues.

### 3.2.4 Microfine Cements

This chapter describes application of “microfine” cements system as possible blocking agent. Cementing using recently developed engineered micro-cement slurries allows small channels to be efficiently filled with cement materials, developing a permanent, high quality seal with high compressive strength and low permeability.

Conventionally the development of micro-fine cement had a lot of practical use in near wellbore treatments for casing repair, blocking fractures and channels. The recent development of cement system considered to have potential for deeper placement applications.

#### Cement hardening kinetics and chemistry

The general mechanism of cement setting is hydration process, the chemical reaction when anhydrous cement is mixed with water producing hydrates that are not water-soluble. The curing process causes slow hydration and crystallization process, the interlocking of the crystals gives cement its strength.

#### Packing Volume Factor (PVF)

The main feature of micro-cements introduced in 1980's was the particle size with maximum particle size 10 to 12 micrometers and a median diameter of 6 to 8 micrometers or less. This micro-cement is simply Portland cement that has been ground to a finer size, generally 3 to 10 times smaller than common oilfield cement. However, the smaller size is not a only requirement for cement in order to have deeper penetration. Even micro-cements can be limited in penetrating due to surface charge interaction, that was continuously proven by field experience and research work (Keese et al., 1999).

The new approach for micro-cements is engineered micro-cement (EMS). The packing volume factor (PVF) method separates engineered slurries from conventional slurries. Engineered micro-cement incorporates a particle size distribution that allows a deeper penetration without bridging. EMS slurries are based on the concept of increasing the packing volume fraction of the solids to enhance slurry performance. Increasing the PVF is achieved in the EMS slurries using an engineered particle size distribution, smaller than the cement material. A properly selected particle size distribution allows the smaller particles to fit inside the void spaces of the larger particles (Farkas et al., 1999). An example of bridging and fingering of conventional micro-cement and behavior of engineered deep penetrating slurry is shown in Figure 3.7.

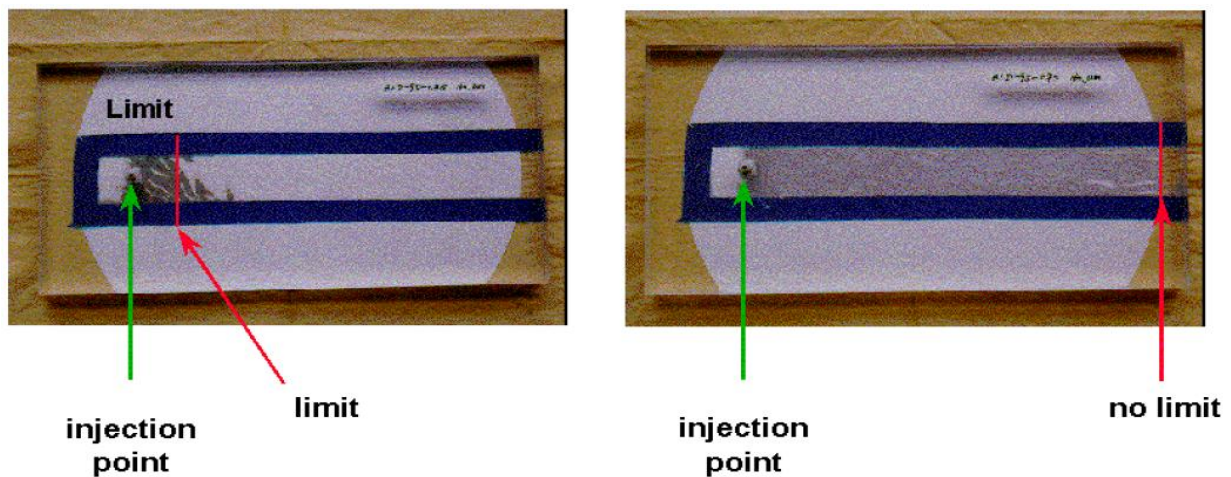


Fig. 3.7 - Comparative injection of micro-cement and engineered deep penetrating slurry into a 160 micrometer slot. (Schlumberger SqueezeCRETE, 2008).

#### Properties of cement

Maximizing the packing volume factor means that there is a higher solids to liquid ratio in EMS slurries when compared to conventional oilfield and micro-cement slurries. This process also creates more stable slurry. Free water in typical EMS designed slurry is zero.

By nature cement exhibit much greater physical stability comparing to any other plugging products and used for high permeability channels plugging when the conventional gel systems not able to withstand high pressure difference. The extensive laboratory research done for evaluation of thermal stability by constant high temperature exposure and temperature cycles for high temperature application, showing the stable properties at temperatures up to 400 °C (Pershikova et al., 2010).

#### Cement potential for thief zone isolation

The first evaluation of possible product for long time reliable isolation of thief zone considered the microfine cement as potentially best candidate for such treatment. The physical properties and thermal stability of cement systems is the most robust and strong isolation and could provide the same properties as a natural cap rock if placed in area over continuous bitumen.

However, the main drawback of system is questionable injectivity for wide lateral placement over bitumen area. The small particle size in order of several microns has a potential for deeper injection, but the possibility of blocking the pore throats and consequent bridging should be evaluated.

### 3.2.5 Foams

Gas/liquid foams offer an alternative to polymers for providing mobility control in chemical floods and have been both proposed and field tested for steam-floods. Its primary targets have been matrix (unfractured) reservoirs (Sydansk, 1994).

Almost all cases of steam flooding have common problems with controlling the mobility of steam and the solution found to the steam channeling is decreasing the mobility in the steam pathways by introducing a non-condensable gas and a foaming agent (surfactant) in the reservoir.

The use of foams to improve the mobility ratios of oil displacing agents arose from laboratory work in the 1950's and 1960's (Bond and Holdbrook, 1958; Fried, 1961; Bernard, 1963). Bernard and Holm (1964) published results in which gas permeabilities in the presence of foam were less than one percent of the permeabilities in the absence of foam.

The paper from Eson and Cooke (1989) gives analysis of field experience and pilot tests of foam systems as diverting agents and discusses the possibilities for different application methods.

#### Chemistry of foams

Foams are special kind of colloidal dispersions of gas bubbles in liquids. Gas liquid dispersions are normally unstable and usually will break in less than one second. If surfactants are added to the liquid, stability is improved greatly so that some foams can persist indefinitely.

Foaming surfactants consist of molecules having a hydrophilic group attached to a long hydrophobic tail. They are classified according to the nature of hydrophilic group. Surfactant molecules orient themselves so that the hydrophilic group in an aqueous environment, and hydrophobic tail is in a non-aqueous environment. They will, therefore, concentrate at the liquid-gas interfaces in foams. Not all surfactants will act as foaming agents some will destabilize the foam structure and can be used instead as defoamers.

Different foaming agents used for formation of stable foams depending on specific application. In oil field applications the foam commonly generated in reservoir by first injecting a foaming surfactant and followed by gas (nitrogen) injection. The foaming process in reservoir is generally controlled by amount and speed of injected gas.

### Steam diversion by foam

Foams can reduce the mobility of a gas phase drastically. The mobility reduction caused by foam can be viewed as an increase in a single-phase viscosity or as a decrease in the gas phase permeability. The low foam mobility in permeable media flow is postulated to be caused by at least two different mechanisms: (1) the formation of or the increase in a trapped residual gas phase saturation and (2) a blocking of pore throats caused by the gas films (Kabir, 2001).

The formation of high viscosity lamella provides drastic reduction of gas phase mobility, the relative permeability reduction is shown in Figure 3.8.

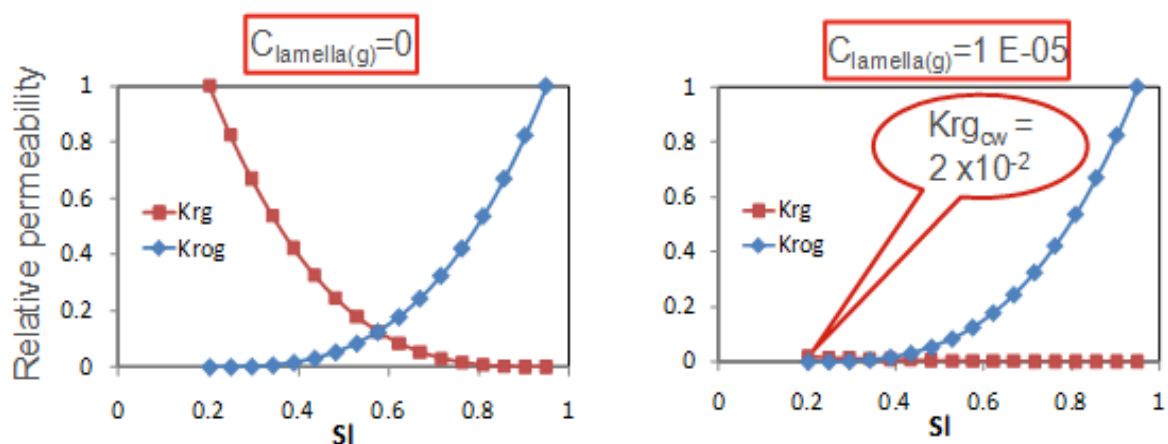


Fig. 3.8 - Gas phase permeability depending on lamella concentration (Thebault et al., 2011).

### Foamed gel formulation

Foamed gels also used in plugging high permeability streaks in waterfloods (Miller and Fogler, 1995). In-situ generated foamed gel barrier can cause a long term waterflood diversion. It provides an intermediate degree of plugging, yet requires substantially less polymer than bulk gels. However, foamed gel barriers break down when the pressure exceeds a critical value. This critical pressure that initiates breakdown varies with foam quality, permeability and gel properties.

A foamed gel consists of a polymer crosslinking solution containing a surfactant that is foamed using a gas. A foamed gel is created by means similar to those used for aqueous foam generation. The major difference between foamed gels and aqueous foams is that the external phase of the foamed gel crosslinks, greatly enhancing the mechanical stability of the foam system. Aqueous foams, whose interfaces are stabilized by surfactants alone, collapse if surfactant free water flows through them. As a result, aqueous foams are not applicable for

long term waterflood diversion treatments. Ideally, the polymer gel foaming solution would not gel during the injection of foam into the porous matrix. Thus, a foamed gel system behaves like an aqueous foam during injection, and after gelation, it behaves in a similar fashion to a gel.

### Properties and stability of foams

Generally the foam can be characterized by its quality and texture, which are depend on chosen foaming surfactant as well as mechanism of foam formation.

Foam quality depends on its composition, that is, of the relative amount of gas and liquid in the foam. Foam quality at certain operating conditions can be obtained from the following expression (Forgiarini and Salager, 1993; Hutchins and Miller, 2003)

$$x = \frac{V_G}{V_G + V_L} \quad (3.2)$$

where  $x$  is the quality and  $V_G$  and  $V_L$  are the gas and liquid volumes at the operating conditions, respectively.

Foam texture refers to the bubble size distribution of the dispersed gas phase. Qualitatively, foam texture can be described as fine texture (small bubbles) or coarse texture (large bubbles) and as homogeneous or heterogeneous, that is, comprised of similar or dissimilar size bubbles.

In reservoir conditions the behavior and stability of foam is dictated by surfactant type, concentrations also the reservoir fluids and properties such as temperature, salinity, etc.

Salinity affects foaming surfactants depending on electrolyte type and concentration. For example, high electrolyte concentration may improve foam stability as the double layer repulsion is increased (Verwey and Overbeek, 1948). Conversely, a high ratio of calcium to sodium ion concentration increases surfactant loss. With relatively high concentration of surfactant the effect of salinity is negligible, but becoming more pronounced at lower surfactant concentrations.

The instability problems of foams in high oil saturation is well known effect, hence the main target for foam formation is areas already swept or initially with low oil concentration.

As the primary application of foam systems is steam diversion, numerous papers and experimental research done for thermal stability evaluation of foams (Hirasaki, 1989; Friedmann et al., 1991) showing stable behavior at temperatures higher than 200 °C. Maini



and Ma (1986) also studied the foam stability at elevated pressure and temperatures up to 200 °C, concluding the increased foam stability with increased pressure.

#### Foam injection potential for thief zone isolation

The well known fact that the best temperature isolation is air could be effectively applied on thief zone isolation, providing best solution for steam chamber temperature isolation from overlying water and gas zones.

The existing experience with steam diversion would serve as a good technical background for designing such treatment in Surmont area. The reduced mobility of vapor phase by foam systems seems to be more effective treatment rather other solid blocking agents, which are able to block steam, but still serve as temperature conductive material.

Despite of the all attractive properties of foam systems there are few drawbacks of foam systems potentially restricting their application as blocking agent in SAGD operations.

The lack of yield strength is one of the most concerning point, as the pressure difference between injected steam and overlying thief zone may displace the foam plug from initial placement area. The long term stability of foam isolation in such conditions is questionable.

The mentioned instability problem upon contact with oil to be verified, as the steam chamber by nature of SAGD process contains a lot of volatile oil products as result of steam constant mixing and heating continuous bitumen area.

### **3.2.6 Simple inorganic salts**

The potential isolation by simple inorganic salts precipitation in reservoir refers to scaling problem solution. In fact, such isolation technique is opposite task to solving the scaling issues. The desired permeability reduction is assumed to be achieved by precipitation of salt in thief zone layer by different mechanisms (low solubility at high temperature, mixing of incompatible waters, etc.)

#### Precipitation mechanism

Calcium sulfate is the most common salt causing scaling problems in reservoir and commonly in area near wellbore due to injection and mixing two incompatible fluids in porous media. The example could be North Sea operations where seawater injection is common practice.

Yuan and Todd (1991) studied possible techniques to predict sulfate scaling tendency in oilfield operations. Based on coreflooding experiments Tahmasebi et al. (2007) described the possibility to predict permeability reduction due to calcium sulfate scale formation. Another laboratory study was done by Abu-Khamsin et al. (2005) to develop a methodology for the determination of the reaction rate of scale precipitation in porous media.

All laboratory evaluations confirm the increased rate of CaSO<sub>4</sub> precipitation at higher temperature, whereas pressure had only slight effect. This fact is explained by lower solubility of calcium sulfite at elevated temperature, which will precipitate upon heating partially blocking the porous media. Figure 3.9 shows the solubility of CaSO<sub>4</sub> depending on temperature.

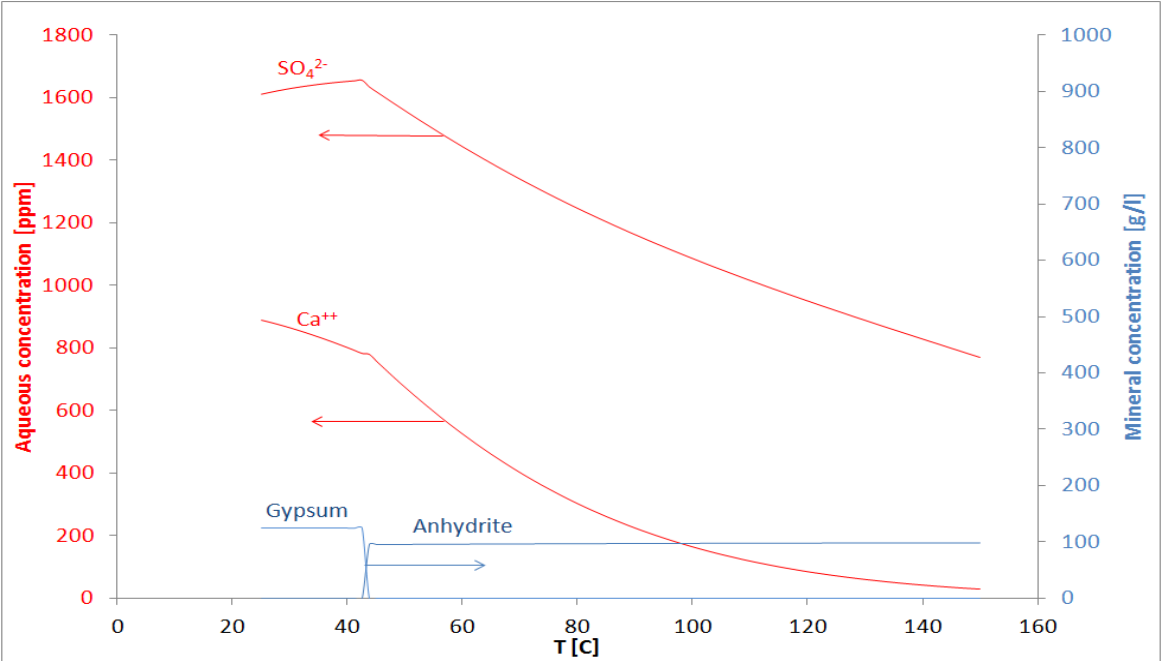


Fig. 3.9 - Solubility of calcium sulfate vs. temperature (Levitt et al., 2011).

The experimental research done by Tahmasebi et al. (2007) on core samples describes permeability reduction by changes in Apparent Mobility Ratio (AMR):

$$AMR = \frac{(k/\mu)}{(k/\mu)_i} \tag{3.3}$$

where  $(k/\mu)_i$  initial mobility and  $(k/\mu)$  mobility at the any time. The results of experiment shown in Figure 3.10 with measurements of AMR done versus pore volumes of saturated saline solution injected in the sandpack at temperature 70 °C.

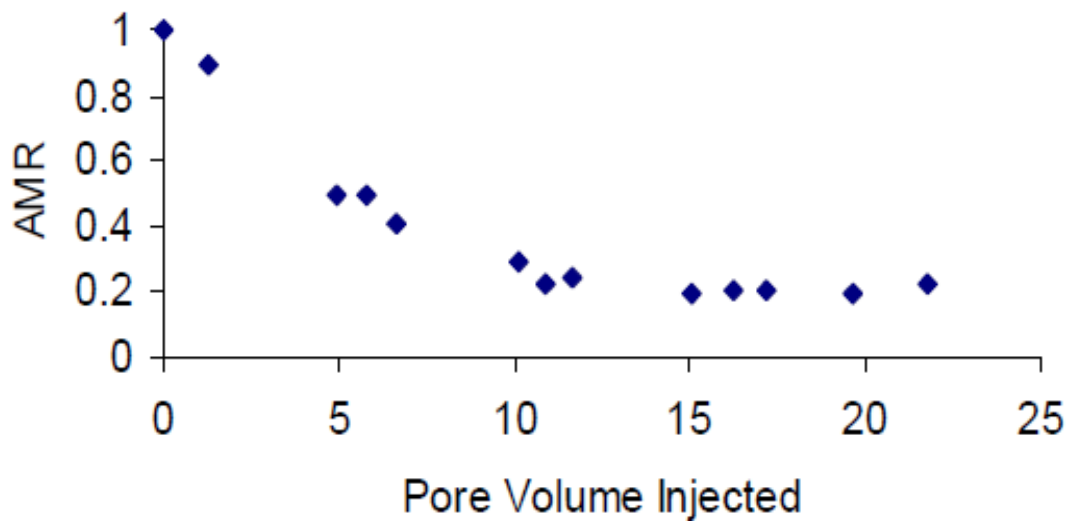


Fig. 3.10 - Variation of AMR versus calcium sulfate injection at 70 °C (Tahmasebi et al., 2007).

Simple inorganic salts potential for thief zone isolation

The low solubility at high temperature considered to have potential in thief zone isolation by formation of insoluble precipitates upon heating due to development of steam chamber. The idea would be inject super saturated brine in overlying water zone which would start precipitate when the temperature start to rise and have enough permeability reduction when the actual steam chamber arrives. With such setting mechanism the steam blocking would occur only in areas where it is actually have tendency to breakthrough.

The injectivity of such super saturated saline solution would be water like, but the wide lateral placement of such solution would possibly lead back to scaling problems forming during the injection, which would possibly restrict the large volumes to be injected in the thief zone areas.

The placement of sufficient concentration of salt in target area is also questionable due to initial low solubility of salt, possibly large amount of brine should be injected in target areas to achieve desired saturations.

Another concern is possible not efficient final permeability reduction even if the precipitation mechanism would work as planned as the Surmont reservoir primarily consists of unconsolidated sand with high degree of heterogeneity, which would be possibly not a best candidate for such plugging method.

### 3.2.7 Hybrid systems

The common practice in chemical isolation treatments is mixing compatible chemicals in order to obtain desired properties. This applies to all conventional products as cements, silicate and polymer gels, foams, etc.

The paper from Burns et al. (2008) describes specific formulation of mixed silicate and polymer systems for development of efficient casing repair gel, which could be formulated as soft water shut-off gel or ringing rigid gel for casing leak correction.

Lacatos (1999) gave an overview of possible mixing of silicate systems with polymers on Hungarian field, concluding that such combination allow to eliminate most of the drawbacks of original pure silicate systems.

Possibility of mixing the acrylic and epoxy emulsion for thief zone plugging discussed by Knapp and Welbourn (1978). The developed gel systems claimed to be not sensitive for shear degradation, high temperature stable (300 °F) and resulting in tough, rubbery acrylic/epoxy plastic material.

#### Hybrid systems potential for thief zone isolation

The hybrid systems also considered to be possible solution for thief zone isolation application if formulation and compatibility of chemicals allow development the final product addressing the main needs of such application.

For specific thief zone isolation the hybrids systems have potential to provide higher injectivity of desired chemicals, in order to achieve deep placement of agents. The compatibility of different chemicals, possible negative effect on isolation kinetics and other parameters to be evaluated in order to find potential effective hybrid solution.

The practical solution of hybrid systems would be the possibility to reduce the high cost chemicals. The economic concern with chemicals having desired properties, but very high cost to inject required volumes for isolation, could be solved if a hybrid system is developed so the promising product can be injected for thief zone isolation in cost effective manner.

### 3.3 Preliminary screening results

The main chemical groups evaluated as having potential for a thief zone shut-off are:

1. Silicate gels
2. Polymer gels (crosslinked rigid polymer gels)
3. Resins (Epoxy)
4. Foams
5. Cements
6. Simple inorganic salts

Based on the literature review presented earlier and the communications with relevant experts the preliminary screening chart (Table 3.1) was developed ranking the evaluated products by their potential to meet the required shut-off criteria. In this table, the various products are ranked in a decreasing order (i.e. from agents with minimum technical restrictions for thief zone isolation to the ones having low potential and serious limitations for such treatment).

Based on preliminary screening results, the epoxy resin, silicate and polymer gels are chosen for further consideration for experimental screening in order to evaluate the critical parameters and potential for thief zone isolation. Cement systems, inorganic salts and foams are eliminated from further consideration evaluated as having serious drawbacks and technical difficulties for such treatment.

The epoxy resin system is evaluated to have highest potential for thief zone isolation and steam shut-off as the product having minimum technical limitations. Having good isolation and thermal stability properties, the injectivity of naturally viscous epoxy considered as important subject for further experimental evaluation.

The aqueous gel systems as silicates and polymers having a good isolation and injectivity properties with numerous field applications should be evaluated for thermal stability as the high temperature conditions may seriously reduce their long term stability and permeability reduction ability. Between these systems, the silicates assumed to have higher thermal stability comparing to polymers, due to polymer's specific chemical reaction at high temperature as hydrolysis.

Table 3.1 - Preliminary screening chart of potential products for zonal isolation by key parameters.

		Permeability reduction	Thermal stability	Transport/injectivity ( <i>viscosity, shear stress, particle size, adsorption</i> )	Negative interaction with resident water/gas zone ( <i>chemical</i> )	Negative interaction with steam front compound ( <i>chemical</i> )
1	Epoxy resin	Effect of residual water and solvent evaporation should be evaluated	High	Naturally high viscosity, evaluate possibility mixed solution (e.g. micro emulsion)	Likely stable	Possible, but no indications
2	Silicate	High permeability reduction is possible	Possible lowered permeability reduction with time ( <i>gel shrinkage</i> )	Good injectivity, <i>need to manage gel kinetics</i>	High sensitivity for divalent cations with subsequent precipitation	Possible dissolution if exposed to high pH
3	Polymer	High permeability reduction is possible	Sensitive to high temperature ( <i>hydrolysis process</i> ) as well as gel shrinkage	Good, <i>need to manage gel kinetics</i>	to be investigated	to be investigated
4	Injectable Cement	High	up to 300-400 °C; Possible thermal expansion	Possible restriction on injectivity due to petrophysical parameters	Not critical	No
5	Salts	Permeability reduction may not be sufficient	Precipitation upon heating; low solubility at high temperature	High	to be investigated	Possible dissolution and migration
6	Foam	No yield stress	Generally low thermal stability of common surfactants	High	Not likely	Destabilization in presence of oil component

### Limitation of cement systems

The proposed microfine injectable cements even having best physical strength, isolation and thermal stability and small particles (up to 2-3 microns), considered to be not able to propagate to long distances in reservoir without plugging the pore throats and eliminated from further consideration.

The particle-to-pore diameter ratio controls the formation permeability reduction. The Hagen-Poiseuille flow model, which approximates the porous media as a bundle of capillary tubes, can be used to estimate the average pore radius (Hirasaki and Pope, 1974) as follows:

$$r = \sqrt{\frac{8k}{\phi}}, \quad (3.4)$$

For Surmont field the pore size of unconsolidated sand estimated to be in order 4 to 5 micron with permeability 1 Darcy and porosity 30 to 40%.

From capillary tube model with adsorbed layer thickness,  $d$ , the following relation is:

$$RRF = \left(1 - \frac{d}{R}\right)^{-4} \quad (3.5)$$

Where  $R$  is the pore radius and  $d$  is particle size. By plotting the pore-to-particle diameter ratio versus RRF (Fig. 3.11), the qualitative formation damage rule can be applied (Stavland et al., 2011b).

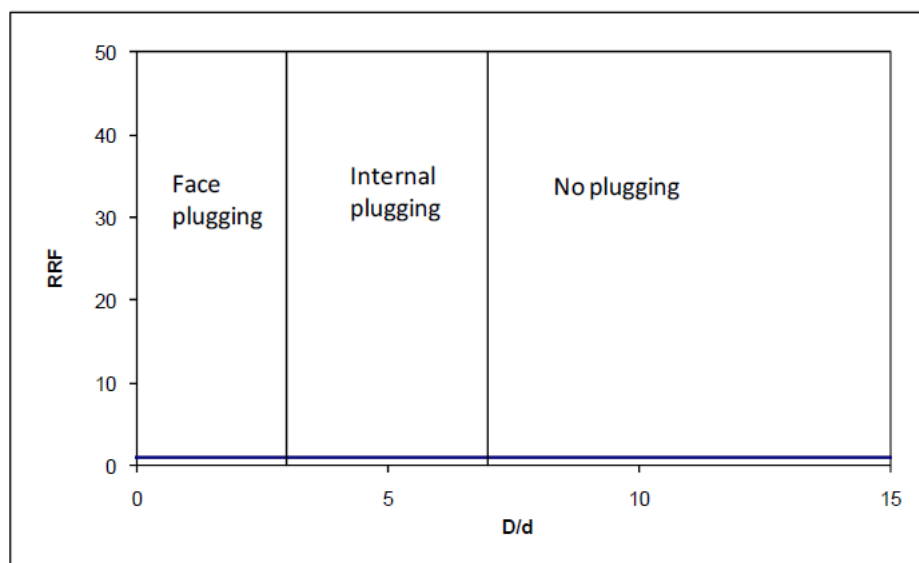


Fig. 3.11 - Permeability reduction versus pore-to-scale ratio (Stavland et al., 2011b).

Based on rough calculation, the particle size of injected agent should be 7 times smaller than calculated pore throat size (4-5 micron) to be able flow through porous media without plugging. Hence the required particle size should be 0.5 micron and less. That requirement is not met by even finest available cements for Surmont field conditions.

#### Limitation of inorganic salts

The idea of blocking the porous media by  $\text{CaSO}_4$  precipitation due low solubility at high temperature evaluated to have restriction of maximum saturation of injected brine by the salt.

The solubility of calcium sulfate is 2.1 gram in 1 liter of water. The concerned restriction is that the injected super saturated brine would not be able to have enough concentration of salt to effectively block the porous media.

Taking into account the limitations on brine saturation, the potential blocked volume assumed to be very small if the salt is precipitated from the solution at elevated temperature. The higher plugged volume is achievable only if the large volumes of super saturated solution are continuously injected through the porous media, which would be not possible for the case of thief zone isolation application.

#### Limitation of foam systems

The permeability reduction agents as silicate or polymer gels have substantial strength once formed in reservoir and have certain yield strength before the gel is displaced or broken. The foam systems in reservoir condition often used as steam diverter due to favorable relative permeability relation, but having no yield strength. The foam layer considered to be displaced further in reservoir due to arising pressure gradient from continuous steam injection during the SAGD operations.

The literature describes hundreds of successful applications of foams as steam diverter, allowing steam to access the unswept layers, but its potential for steam shut-off over wide area under high pressure difference is evaluated as very low.

However, foam solution could be practical for Surmont case if additional strength is achieved by foaming the gel or resin solution so that uniform foam structure formed with enough physical strength. Another practical solution by foam is possibility to reduce the cost of injected materials if the desired product for steam shut-off is identified, but not economic to inject as continuous layer.



## 4. Experimental work and results

In this section the experimental work is presented for the products evaluated to have potential for successful thief zone treatment. The screening experiments done based on developed criteria for thief zone application and potential technical drawbacks of chosen products.

Key parameters for thief zone shut-off application:

- Controlled delayed gelation
- High permeability reduction
- Thermal stability (200-250 °C)

Based on the experimental results obtained for each system, the product applicability is evaluated for the Surmont case.

### 4.1 Experimental evaluation of silicate gels

This section describes the experimental work done with silicate gels in order to evaluate the general properties as gelation time and permeability reduction, as well as screening experiments on thermal stability of silicate gels.

In following experimental work commercial sodium silicate from Sigma-Aldrich is used with SiO<sub>2</sub> concentration at 27,84% and SiO<sub>2</sub>:Na<sub>2</sub>O molar ratio 3,4. The initial pH of sodium silicate solution is 11. Diluted 2 mole hydrochloric acid was chosen as activator for gelation process by lowering the pH value of silicate solution.

#### 4.1.1 Bulk experiments

##### Gelation kinetics at room temperature

Bulk experiments were done in order to evaluate the general gelation kinetics and pH dependency of silicate solutions. The gelation is inspected visually and degree of gelation reported according to gel coding developed by Stavland (2011a) (Table 4.1).

Table 4.1 - Gel coding.

Gel code	Description
0	Clear and low viscous fluid
1	Cloudy and low viscous fluid
2	Cloudy and high viscous fluid
3	Rigid gel

Effect of pH on bulk gelation time is studied by adding different amount of 2M HCl to 4 wt% sodium silicate solution and the time of gel reaching code 1 is reported (Table 4.2). The 50ml solution samples prepared diluting the sodium silicate in distilled water.

Table 4.2 – 4 wt% silicate solution with different amount of activator added.

	1	2	3	4	5	6
wt% of 2M HCl	8,5	9,0	9,5	10,0	10,5	11,0
pH (after mix)	10,46	10,43	10,32	10,29	10,1	10,07
gel code 1, hours	11,3	8,8	6,5	4,0	2,3	1,0

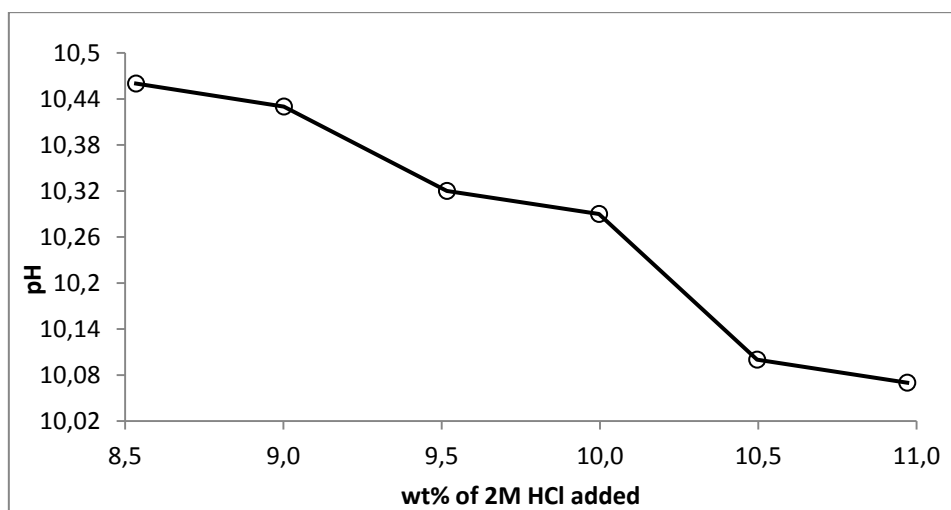


Fig. 4.1 - pH of solution after mixing – T=20 °C.

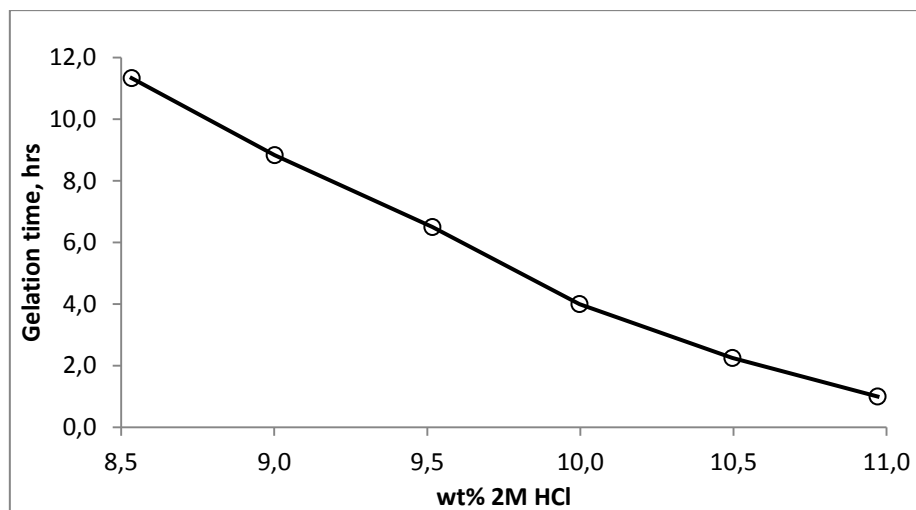


Fig. 4.2 – Silicate gelation time versus amount of activator –  $T=20\text{ }^{\circ}\text{C}$ .

The bulk gelation experiments on silicate gels result in opalescent, ringing and brittle structure once reached the gel code 3. Results of pH measurements right after mixing (Fig. 4.1) and gelation time measurements versus amount of activator added (Fig. 4.2) are in agreement with previous laboratory evaluations (Stavland et al., 2011a). The amount of HCl added could be good controlling measure of pH reduction and consequent gelation kinetics.

#### Bulk thermal stability

After the gel samples with 4 wt% silicate reached the gel code 3, the closed bottles with formed gel set in oven for  $80\text{ }^{\circ}\text{C}$  to qualitatively evaluate the effect of elevated temperature and water expulsion (Fig. 4.3).



Fig. 4.3 – 4 wt% silicate gel samples heated in oven at  $80\text{ }^{\circ}\text{C}$ .

The gel samples heated up for 1 week to 80 °C showed good stability without signs of gel degradation, although small water expulsion is noticed in all heated samples.

The gel shows good stability up to water boiling temperature, at temperatures higher than 100 °C the vapor phase formed in gel structure destroys the gel very fast.

#### **4.1.2 Beaker experiments with sand**

##### **Thermal stability**

##### **Experiment 1**

Experiments done with two different types of sand (coarse and fine grain) mixed with activated 4 wt% silicate in order to evaluate the bulk thermal stability of gelled samples by heating the gelled sand samples in oven. The 300 ml of sand used in each experiment with 100 ml of activated sodium silicate solution (Fig. 4.4).



Fig. 4.4 – Fine grain sand consolidated by silicate gel.

The open gelled sample left for heating at 150 °C for 2 days, this resulted in sand drying out from top to bottom. The sand after heating seems to have cemented structure with hard, rock-like outer layer (Fig. 4.5).

Similar behavior is noticed with coarse grain sand experiments. The gelled sample of coarse sand resulted in hard cemented structure after heating in oven at 150 °C.



Fig. 4.5 – Fine grain sand dried out and consolidated after heating at 150 °C.

### Experiment 2

The effect of heating the gelled sand samples by vapor is tested in the following experiment. The fine grain sand and 4 wt% silicate used for preparing the gelled sample in open end beaker (Fig. 4.6).



Fig. 4.6 - Gelled fine grain sand in open end beaker.

The exposure to steam and effect of heating by steam on sand consolidation is evaluated by simple experiment shown in Figures 4.7 and 4.8.

The vapor developed by simply boiling water and the open end is connected to beaker with gelled sand. Later small net is placed on the bottom of gelled sand column, so that steam still access the sand, but the falling apart of sand is prevented.

The heating continued for 6 hours and resulted in gelled sand losing its consolidation with mud-like consistency in area of contact with vapor, while the rest of gelled sand on top left intact.

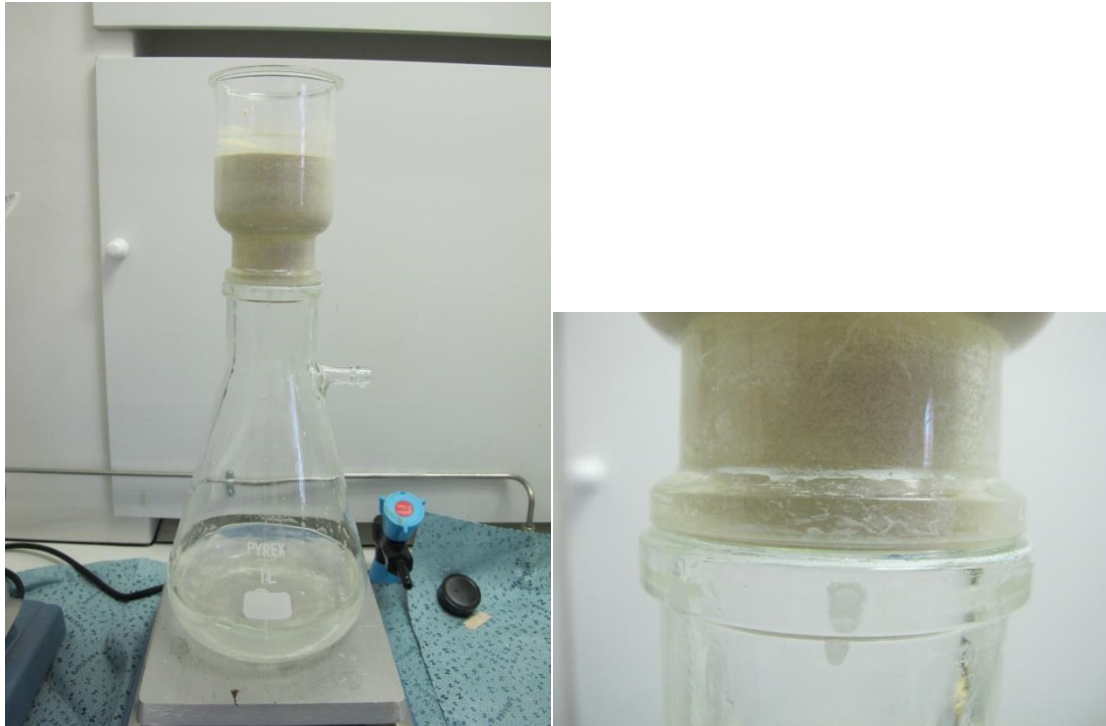


Fig. 4.7 - Installation for heating the gelled sand sample by vapor from boiling water



Fig. 4.8 – Gelled sand sample before and after heating by vapor.

### 4.1.3 Sand column experiments

The static experiments performed on sand columns filled with sand and plugged with silicate gel in order to evaluate permeability reduction and study effect of high temperature on residual permeability reduction. The sand column filled with coarse and fine grain sand, the

column filling with sand technique allows achieving different initial permeability. With high vibration and slow filling, the coarse sand results in permeabilities from 10 to 20 Darcy, when the fine grain sand almost always results in permeability 0.8 Darcy.



Fig. 4.9 - Sand column installed in oven.

The injection of water in gelled column can be done in two ways:

- Constant flow rate injection with measurement of pressure drop in sand column
- Injection with constant pressure with measurement of effluent flow rate

Before silicate solution injection, distilled water continuously injected in sand columns and then the permeability measured at flow rates of 1, 2 and 4 ml per minute.

The pore volume of each sand column calculated by injection of 2g/l KI solution and followed by UV measurement at core effluent. The calculated pore volumes of tested sand columns were from 110 to 140 ml.

#### **4.1.3.1 Permeability reduction at room temperature**

##### Experiment 1

The possibility of delayed gelation of sodium silicate, which could be activated and form rigid gel by thermal front in reservoir is tested by injection of 4 wt% silicate solution with low

amount of activator (3 wt% of 2M HCl). The 3 pore volumes of activated silicate solution injected with flow rate 5ml/min and followed by sand column heating up to 80 °C to speed up the gelation. Effluent samples also heated and gelation monitored visually.

The effluent samples did not form rigid gel (Fig. 4.10), resulting in solution with flowing gel aggregates.



Fig. 4.10 - Effluent after injection and after 4 days heating in oven at 80 °C.

Distilled water injected with constant flow rate (1ml/min), resulting in short pressure build-up and then continuous decrease (Fig. 4.11), which most likely indicate weak gel breakage by injected water.

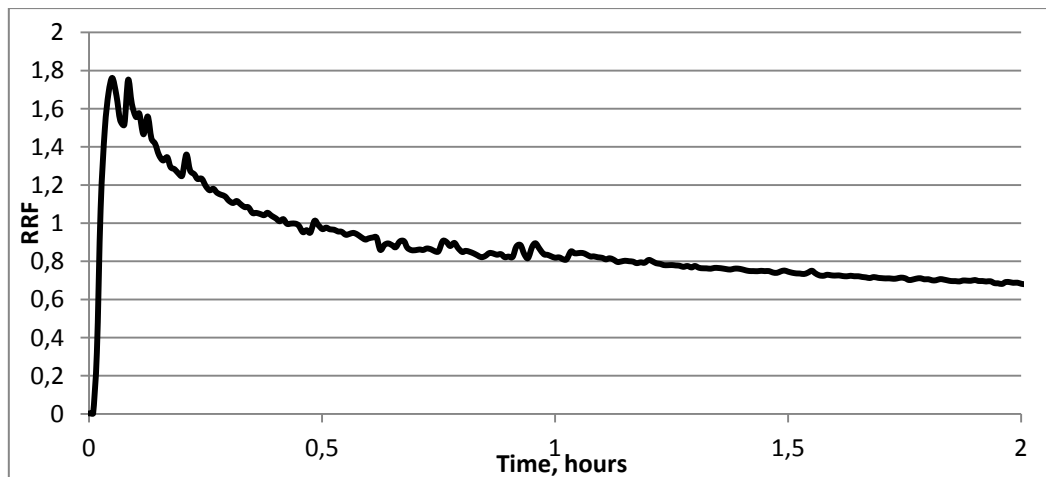


Fig. 4.11 - Residual resistance factor versus time for a silica gelled sandpack column.

## Experiment 2

Coarse grain sand used to fill the column with longer filling time and higher vibration, which resulted in permeability 10 Darcy. The 3 pore volumes of activated 4% wt silicate solution



with 9 wt% of 2M HCl injected at flow rate 5 ml/min. Effluent samples and sand column closed and set heating at 60 °C for 5 days when the effluent sample reached gel code 3.

Distilled water injected in gelled column stepwise at constant pressure from 1 to 3 bars (Fig. 4.12). The total pressure difference monitored and effluent flow rate is measured when the pressure difference stabilized.

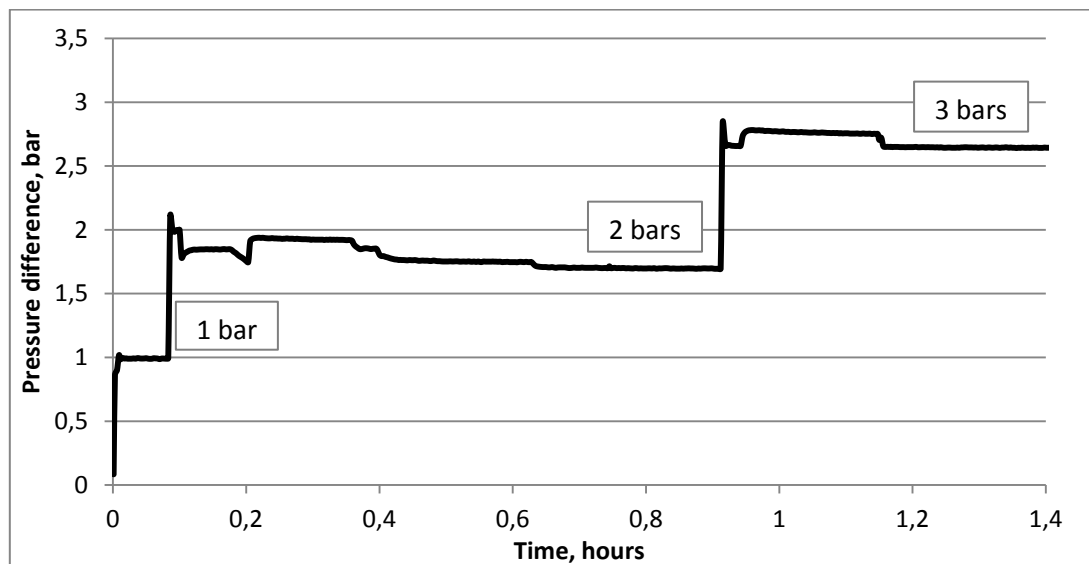


Fig. 4.12 - Pressure build-up in sand column at constant injection pressures 1, 2 and 3 bars.

The effluent flow rate was measured when pressure difference in sand column stabilized for permeability calculations. The permeability of sand column reduced 1000 times, resulting in final permeability 0,01D from initial 10 Darcy.

### Experiment 3

Fine grain sand “Sikaisol” with particles size 80-100  $\mu\text{m}$  used for evaluation of permeability reduction and high temperature effect. The permeability measured after filling the column was 0,8 Darcy. The same concentration of silicate and hydrochloric acid was used as in previous experiment. However, the early plugging is noticed during activated silicate solution injection in sand column with flow rate 5 ml/min. After the 2,5 pore volumes of silicate solution, the sand column plugged. The filled column and effluent left for heating at 60 °C until the effluent samples reached gel code 3 (Fig. 4.13).



Fig. 4.13 - Effluent sample after silicate injection and after heating at 60 °C for 4 days.

Distilled water was injected in gelled sand column at three constant pressures from 1 to 3 bars. Sand column with initial permeability 0,8 Darcy was plugged by silicate gel (Fig. 4.14), no flow occurred during the water injection.

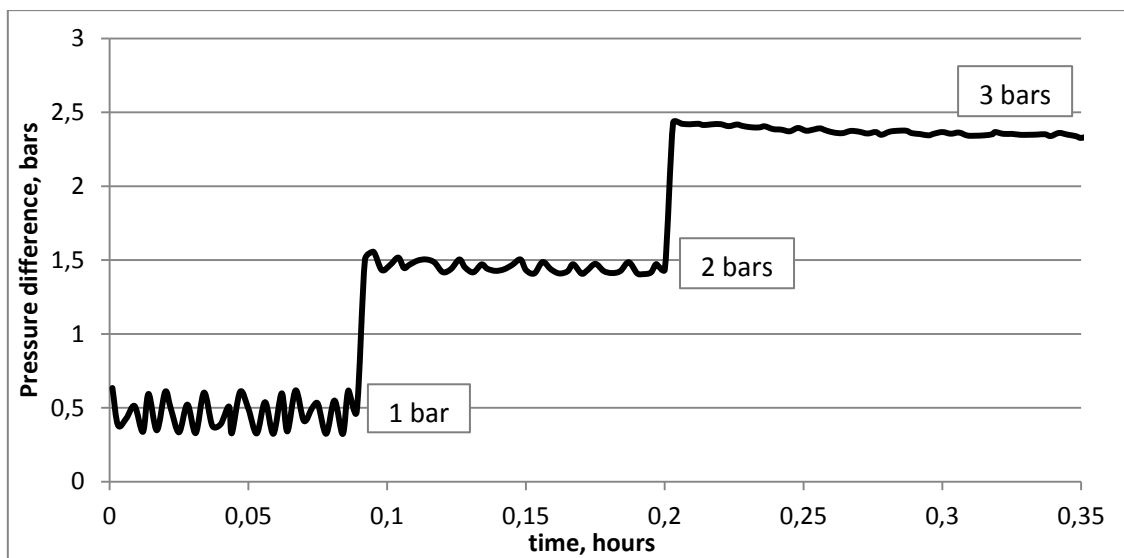


Fig. 4.14 - Pressure build-up in sand column at constant injection pressures 1, 2, and 3 bars.

The gel plugged the column withstand constant injection pressure 3 bars, which is relevant to pressure gradient of 10 bar per meter.

#### 4.1.3.2 Effect of high temperature

To evaluate effect of vapor phase developed in silicate gel at temperature higher than boiling point, the gelled sand columns from experiments 2 and 3 were heated up to 110 °C with open outlet and the effluent measurement during the heating. The heating at higher temperatures

with backpressure was not performed due to safety concerns of too high pressure build-up in the sand column (Fig. 4.15).

Additional experiment is done to evaluate the effect of high temperature on permeability reduction with plugged sand column with initial permeability 0.8Darcy. The gelled column was heated at 90 °C with open outlet in oven and permeability reduction is measured afterwards.



Fig. 4.15 - Sand column placed in oven for heating.

The gelled sand column from experiments 2 and 3 were heated at temperature 110 °C for 3 and 5 days respectively. The heating resumed until the no or small effluent fluid is present. The measured effluent volumes were almost equal to initial pore volumes of sand columns. Permeability measurements with distilled water showed that the sand columns regained their initial permeability after the experiment.

Another experiment with heating the gelled sand column at high temperature (90 °C) for 4 days is done to evaluate the effect of high temperature on permeability reduction if the temperature not exceeding the boiling point of water. The resulting ratio of initial and final permeability is shown in Figure 4.16.

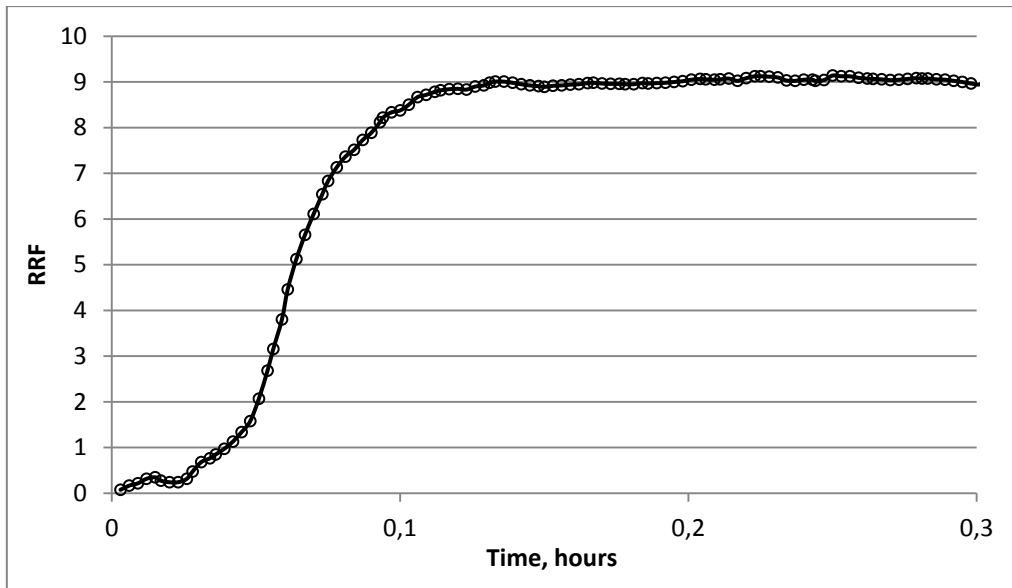


Fig. 4.16 – Residual resistance factor after heating the sand column at 90 °C.

Initially 0,8D sand column was plugged by silicate gel with no flow and heated at 90 °C afterwards. Approximately 20ml of water expulsion is measured during the heating of sand column. The final permeability to water after heating was 0,09 D, hence the permeability of fine grain sand reduced 9 times from initial value.

#### 4.1.4 Analysis of silicate gel applicability for thief zone treatment

The aqueous silicate gels considered as promising isolation agent for following reasons:

- Good injectivity (water-like viscosity)
- Controlled gelation kinetics (silicate and/or activator concentration, temperature, salinity)
- High permeability reduction
- Environmentally friendly and cheap solution

However, the nature of aqueous gel system, initially consisting mostly of water, brought questions regarding stability of silicate gels in high temperature conditions.

##### Gelation kinetics

Existing literature describes in detail the possibility of controlling the gelation kinetics for silicate systems, the mentioned paper from Stavland et al. (2011a) stated the possibility of controlled gelation from minutes to up to a month.

The simple bottle test experiment shows clear correlation of gelation time dependency versus amount of activator added for solution. However, the long term (weeks, month) gelation experiments have not been done, but the possibility of such delayed gelation is evident.

### Permeability reduction

The static gelation experiments with sand columns have shown sufficient permeability reduction for 10 and 1 Darcy sand samples, resulting in 0.01 Darcy and full plugging respectively after gelation.

### Thermal stability

Bulk gelation experiments shown good thermal stability of silicate gel at temperatures up to a boiling point, but easily destroyed at temperatures higher than 100 °C when the developing vapor phase (bubbles) inside the gel break the gel structure.

In sand column experiment the effect of vapor phase beyond boiling point tested by heating gelled samples at 110 °C with open outlet. Repeated experiments showed that the vapor phase formed inside the sand column destroyed the gel structure and displaced all the liquid out column, as result the initial permeability regained after the heating.

However, the heating the whole column with gelled sand inside is not a best representative experiment of heat propagation in SAGD conditions. Ideally it would be gradual heating of gelled sand from the bottom by steam establishing thermal gradient across the gelled sand. In such case the bottom of gelled sand would be zone with highest temperature, and the effect of development of vapor phase inside the gelled structure on permeability reduction would be crucial parameter to make a clear conclusion on silicate gel applicability for thief zone treatment.

The qualitative estimation of gelled sand stability exposed to steam of boiling water is made with open beaker experiment. The experiment with steam heating the area of contact with gelled sand resulted in gelled sand losing its consolidation with mud-like consistency at the end of experiment. The development of vapor phase inside the gel structure is hard to evaluate for this experiment, but the silicate gel seem to be unable to keep sand consolidated when directly exposed to the steam.

### Closing Remarks - Silicate

Silicate gel system is considered to be not applicable for thief zone isolation due to reduced stability of gel at required high temperatures. The development of vapor phase in gelled porous media considered as one of the mechanisms of gel degradation leading to a reduced permeability reduction.

### Remarks on Polymer Gels

The development of a vapor phase is considered to affect also the stability of the crosslinked polymer gels. The additional instability concerns of polymer gels as hydrolysis and precipitation lead to decision to eliminate polymer systems from further consideration.

## **4.2 Experimental evaluation of epoxy resin**

This section presents experimental work done on epoxy resins in order to find efficient solution for injectivity issues and isolation ability at high temperature condition.

Based on developed criteria for thief zone treatment, the identified technical concern for epoxy resin solution is injectivity as the undiluted epoxy has high viscosity (11000-15000 cP), but diluted solutions also available (7-10 cP). Secondary parameters to consider are effect of presence of solvent on hardening kinetics.

In following experiments the EPON 828 undiluted liquid epoxy is used with Versamid 125 hardener from Miller-Stephenson. The mixing ratio of hardener with epoxy used in experiments is 1:3 by weight. The ethyl acetate used as a solvent to dilute liquid epoxy resin.

### **4.2.1 Bulk experiments**

#### Water in epoxy emulsion

The possibility for water in epoxy emulsion is considered as first attempt to lower the viscosity of liquid epoxy. Different amount of water was used for preparation of emulsions. At high water content (>50 wt%) the emulsion became water external (Fig. 4.17).



Fig. 4.17 - Water external emulsion with liquid epoxy.

The 28-35 wt% water content is used to prepare water internal emulsion by slowly adding the water to continuously mixed epoxy. Mixed with hardener, the solutions were split and cured at room temperature and at 83 °C. The samples 35 wt% solution also heated at 150 °C after hardening to examine the behavior of internal water content.



Fig. 4.18 - Epoxy samples mixed with water after hardening.

All the samples formed strong hard material, those which were cured in oven at 83 °C hardened in 24 hours, while hardening at room temperature was about 3 times longer. The samples cured at room temperature did not form stiff hard structure (second sample from left in Fig.4.18).

The heating at 150 °C of sample with 35 wt% water content cured at room temperature resulted in development of vapor phase (bubbles) phase inside the mixture with complete hardening of mixture in 6-8 hours, while the other 35% samples cured at 83 °C did not show any changes after additional heating at 150 °C.

In all experiments the emulsion with internal water phase does not lower the viscosity of solution considerably. Also the emulsions seem to be unstable and start to separate in 1-2 hours.

#### Experiments with solvent

In order to achieve sufficient injectivity, the formulation of epoxy with solvent is studied in bulk experiments with different amount of solvent (ethyl acetate) added to epoxy resin before mixing with hardener. In all experiments with dilution of epoxy, the ratio between hardener and epoxy kept constant (1:3).

The solvent is added to epoxy resin under mixing, after the solution reached homogeneous consistency the hardener is added and mixed again. The viscosity measurements were conducted after the mixing procedures on Brookfield viscometer (Table 4.3 and Figure 4.19).

Table 4.3 - Viscosity and hardening time depending on amount of solvent

Amount of solvent, wt%	5	15	30	40	50
Viscosity, cP (20°C)	1150	154.7	18	7	2,5
Hardening time, hours (20°C)	6	14	21	26	34



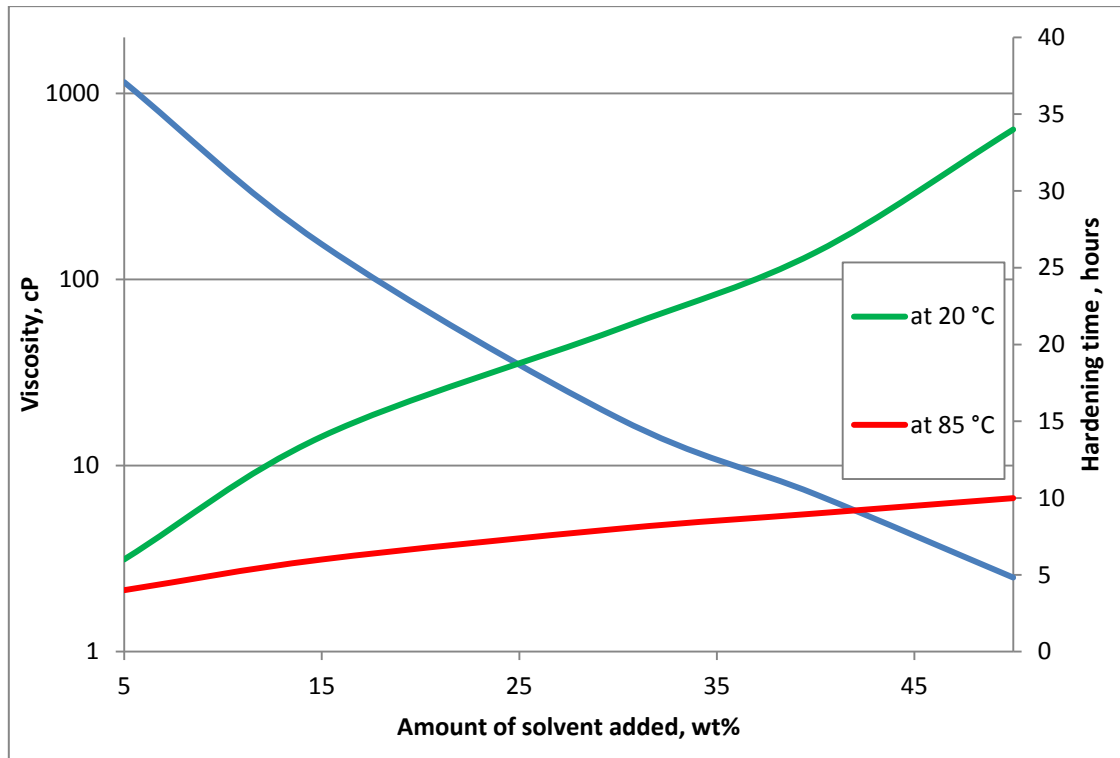


Fig. 4.19 – Bulk viscosity and hardening time depending on amount of solvent added.

All samples split after mixing and also cured at 85 °C in oven, resulting in relatively fast hardening during 4-8 hours (Fig. 4.20).

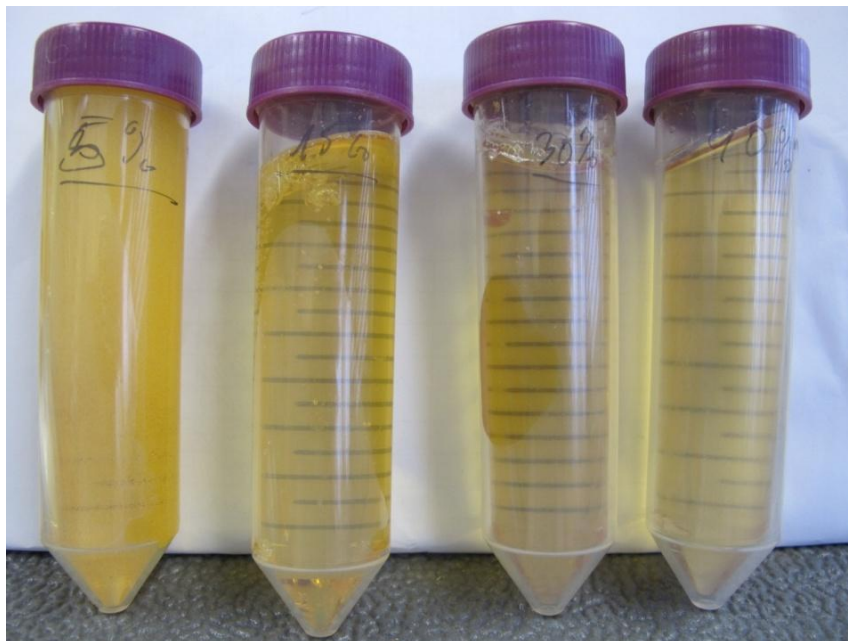


Fig. 4.20 - Samples with different amount of hardener cured at 83 °C.

## 4.2.2 Injectivity experiments

### Experiment 1

Formulation with 40 wt% solvent considered having low enough viscosity (10cP) for injection in a core to verify isolation ability of epoxy solution. Carbonate core is used with permeability 1.5 Darcy installed in PVC cover.

Core preparation and epoxy injection procedures as follows:

- Repeated CO<sub>2</sub> injection and evacuation the core by vacuum ( 3 times)
- Water injection and measurement of pore volume (120ml)
- Water permeability measurement at stable different pressure
- 40% solvent content epoxy solution preparation
- Injection of epoxy solution by buffer column displaced by synthetic oil (MARCOL 52)
- Core left closed with inlet and open outlet at room temperature for 4 days
- Core was heated in oven with gradual increase of temperature up to 110 °C for 7 days

The installation used for epoxy solution injection is shown in Figure 4.21, pump is set outside the installation displacing the epoxy solution by synthetic oil through steel column.

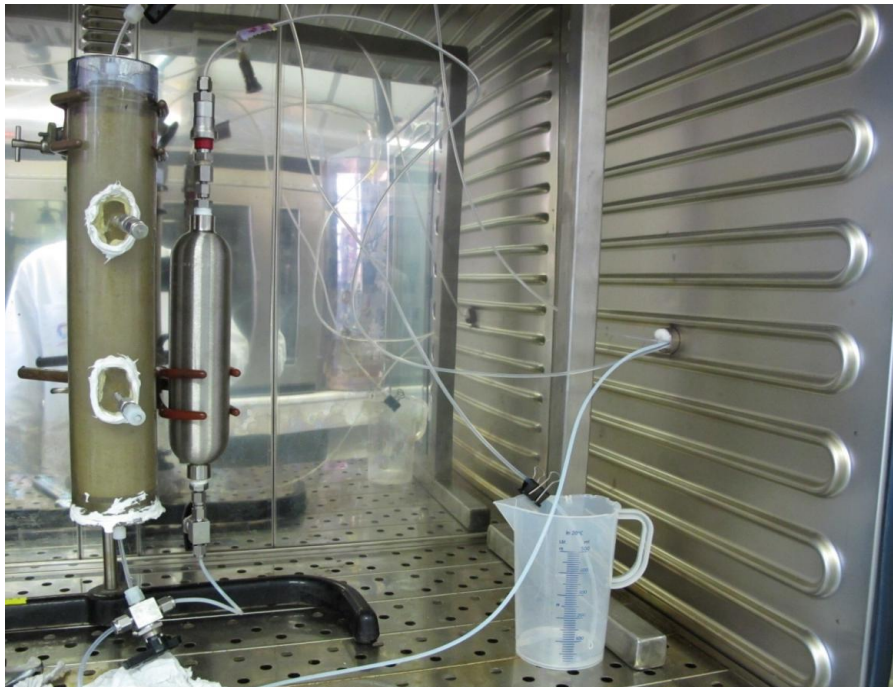


Fig. 4.21 - Epoxy injection in the core with PVC holder.

At least one pore volume is planned to be injected in core initially, but injectivity issues occurred during the experiment. Planned constant injection rate at 1ml/min caused constant pressure build-up to 6 bars, and then the injection with constant pressure (4 bars) is chosen to avoid further pressure build-up. The injection rate stabilized at 0.3ml/min at constant pressure 4 bars.

Core plugging verified by water injection after 4 days of curing at room temperature, then the core set for heating in oven during the week by gradual temperature increase up to 110 °C in order to ensure complete curing of epoxy solution and effect of water/solvent evaporation. The outlet of core was open during the heating to avoid pressure build-up inside the core.

The final permeability to water measurement resulted in 4mD from initial 1500mD.

## Experiment 2

The results of first experiment with injection of epoxy solution to the PVC core lead to assumption that premature hardening happened while preparation and injection procedures. Based on this the MDEA hardener with higher reticulation point is chosen (4 hours at 160 °C) to avoid any premature hardening during the injection of solution.

Simple bulk experiment done before injection experiments in order to verify that diluted solution of epoxy with new hardener will not harden at room temperature, but still able to cure to strong resin when heated.

Bulk experiment procedures as follows:

- MDEA powder hardener is mixed with solvent( ethyl acetate, 40 wt% of total solution )
- The epoxy is added to prepared solution and mixed until clear solution
- The solution split in two samples, one left at 20 °C and second in heated oven at 80 °C
- The second solution heated in oven for 3 days at 80 °C and then set at 110 °C until complete hardening



Fig. 4.22 - Samples of diluted epoxy cured at room temperature (left) and at 110 °C (right).

The samples left at room temperature and heated in oven at 80 °C were still liquid after 3 days. Then the second sample is set for 110 °C in order to verify possibility of hardening the diluted solution. Figure 4.22 shows the second sample formed a strong material, while the first sample was still in liquid form.

The followed injection test is done with 10 micron ceramic filtration disc (Fig 4.23.). The experimental procedures are as follows:

- Measure the permeability the filtration disc to water at three constant rates.
- Prepare 100ml of epoxy solution with MDEA hardener and 40 wt% solvent.
- Measure the viscosity of the prepared solution on Brookfield viscometer.
- Inject solution by displacing the epoxy solution by synthetic oil (MARCOL) at three constant different rates.

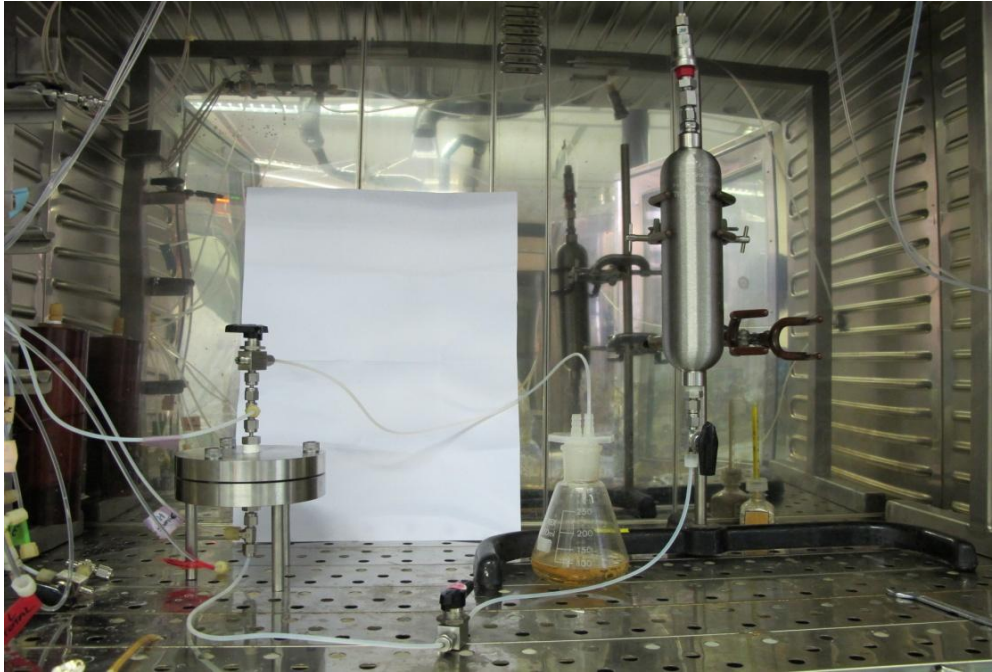


Fig. 4.23 - Installation for injectivity test of diluted epoxy solution.

The permeability to water measured at three constant rates resulted in value 0,4 Darcy. The diluted epoxy solution was injected without plugging at 120, 240 and 480 ml/hour. The pressure difference measured during the experiments shown in Figure 4.24.

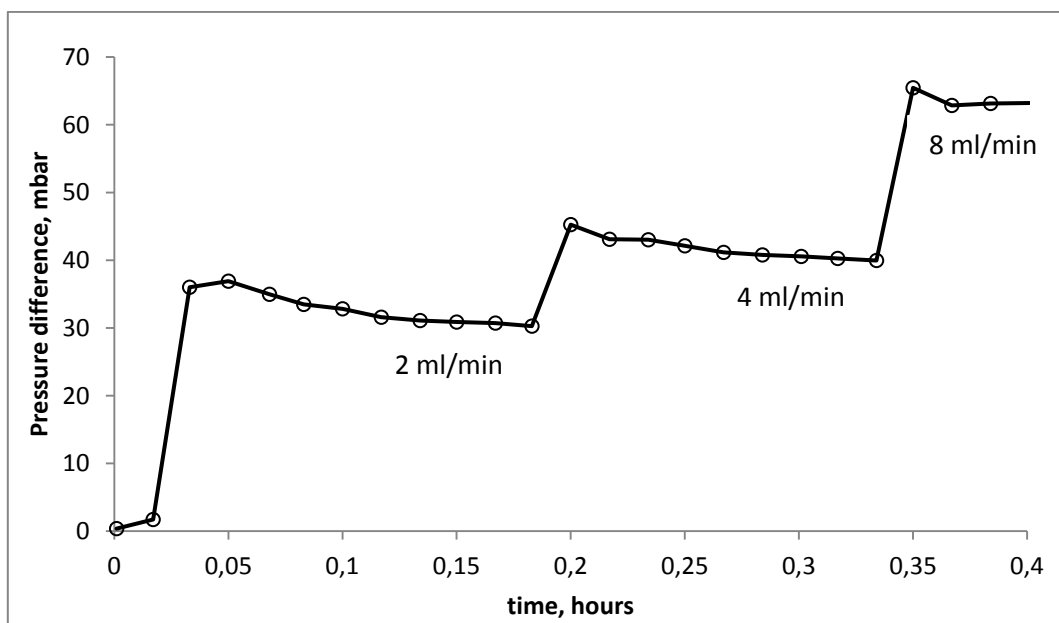


Fig. 4.24 - Pressure difference measured during epoxy solution injection at three constant rates.

Permeability to diluted epoxy solution calculated with account to measured viscosity (7cP) was 0,3 Darcy.

### **4.2.3 Analysis of epoxy resin applicability for thief zone treatment**

Based on preliminary screening the epoxy solution injection is considered as having least technical issues for thief zone isolation. The identified advantageous properties of epoxy resins are as follows:

- high physical strength;
- thermal stability;
- high permeability reduction;
- controlled hardening kinetics (thermosetting ability).

The main drawback of epoxy application identified in screening stage is possible injectivity issues due to naturally high viscosity of epoxy. The screening experimental work is done in order to verify possibility of lowering the viscosity of epoxy using compatible solvent.

#### Water internal emulsion

Possibility of lowering the viscosity by emulsion is checked by preparation of water internal emulsions with epoxy. Despite of the fact that such emulsions were able to harden with water content up to 35%, the viscosity of prepared solutions did not alter from initial epoxy viscosity. Considering that viscosity is the force required to achieve unit flow rate, this behavior explained by emulsion droplets disrupting the streamlines and require more effort to achieve the same flow rate.

#### Dilution with solvent

Experiments with solvent (ethyl acetate) showed possibility of lowering the viscosity of epoxy solution from 11000 to 10-2cP by diluting the epoxy by 40-50 wt% solvent. The samples with high amount of solvent were able to form strong material after curing at room and elevated temperatures.

However, the desired behavior of solution for thief zone treatment would be very long or no hardening at room temperature, while samples prepared in experiments were able to harden during 26-35 hours. Such behavior is explained by reactivity of chosen hardener (liquid Versamid 125), which is able to start reacting with amine groups at room temperature. The hardeners with lower reactivity, which are formulated to react with amine groups at higher temperatures are commercially available.

### Injectivity

The injection of diluted epoxy is attempted with solution mixed with 40% solvent (approximate viscosity 10cP) in order to justify injectivity of epoxy for thief zone treatment.

The low injectivity noticed during the experiment is explained partly by solvent evaporation from the surface of solution and partly by reactivity of chosen hardener (liquid Versamid), which was able to start curing process before the solution is injected in the core.

In next experiment the hardener with higher reticulation temperature is used. The bulk experiment showed no hardening at room temperature, but still able to cure and form strong material when heated at high temperature. The formulation used in injection experiment with filtration disk proving the assumption made, that reactivity of hardener was causing the injectivity issues in experiment with PVC core, because the formulation with new hardener was injected without any plugging at different rates.

### Remarks

Epoxy resin is the most expensive isolation product among the evaluated isolation systems. The use of solvent possibly could reduce the high cost if proper formulation is found, such that maximum diluted epoxy solution still able to effectively harden and shut-off the steam. The preliminary investigation on the epoxy cost indicates that the only economically feasible field application of epoxy is a targeted treatment in areas where the steam breakthrough is most likely to occur with small to moderate sizes of isolation area.

For the estimation of other practical matters, such as availability if the required amount of material, transportation, storage, etc., the epoxy market should be evaluated to ensure that epoxy components are available in required volumes. The possibility for custom-made epoxy should be considered also. The identified early – hybrid systems may be essential way for epoxy application, the possibility for foaming epoxy or reducing the cost of resin by any other method is also an important factor for further consideration of epoxy systems for thief zone treatment.

## 5. Conclusions

A preliminary screening of silicate and polymer gels, epoxy resins, microfine cements, foams and simple inorganic salts as zonal-isolation agents has been carried out to determine the most suitable ones for a further experimental evaluation. The selected screening criteria were based on available literature, agents' technical data, and expert opinions. Silicate gels, polymer gels and epoxy resin systems have been identified as the suitable ones for further investigation. The other agents considered to have serious restrictions for the studied thief zone treatment: injectivity issues for cements, no yield strength for foams, and not sufficient permeability reduction for simple inorganic salts.

Experimentally based evaluation of silicate gels showed that the aqueous gel systems in general are susceptible to degradation and destruction due to vapor phase developing in the gel structure at temperatures higher than the boiling point due to high content of water in gel systems. The results on silicate gels are extrapolated on crosslinked polymer gel systems, which are assumed to have also additional instability issues owing to hydrolysis process at high temperatures.

The epoxy resin systems, identified as most promising and suitable product for thief zone treatment, have to be considered also from a practical point of view if any further research will be done in order to proceed for field application stage.

The experiments with the dilution of the epoxy and the different formulation of hardener were sufficient "concept proof" illustrating a potentially good injectivity of an epoxy-based system. The experimental results showed that the possible injectivity issues identified at screening stage could be solved by a proper formulation of hardener, epoxy and solvent in the injected fluid composition. The wide range of possible formulations gives enough room for designing the viscosity, hardening kinetics and properties of final product for potential application on thief zone treatment.



## **6. Recommendation for further work**

The additional further work required on epoxy resins for thief zone isolation listed as follows:

1. The formulation of epoxy, hardener and solvent should be studied more in order to develop the solution with desired viscosity, hardening kinetics and properties of cured product in core scale experiments.
2. The evaluation of thermosetting feature of epoxy resins should be studied based on field information regarding the temperature gradient development on Surmont Pilot project.
3. The effect of fluid and reservoir properties on water displacement by epoxy solution should be considered in core scale experiments to design effective epoxy placement in desired area.
4. The simulation of potential isolation treatment can be done using the relevant software tools (Eclipse, CMG, etc.) based on existing reservoir models to effectively evaluate the amount and area of placement of isolation product.

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