
Master Thesis

Methods of diverting water-based resins for
sand consolidation



Submitted to the Department of Petroleum Engineering at the Mining
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Thesis

Sehr geehrte Frau Mörtl!

Das von der Firma Halliburton zur Verfügung gestellte Thema der Thesis mit dem Titel

METHODS OF DIVERTING WATER-BASED RESINS FOR SAND CONSOLIDATION

wurde Ihnen zur Ausarbeitung übertragen. Die Arbeiten werden von Ihnen zur Gänze bei der Firma Halliburton unter der Betreuung von Herrn Brad Todd durchgeführt. Die universitäre Betreuung wird von Herrn Univ.Prof.Dipl.-Ing. Dr.mont. Herbert Hofstätter übernommen.

Dieses Schreiben wird nach Abschluss der Arbeit mit eingebunden.

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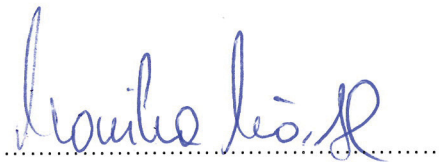
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Glück Auf!



Univ.Prof.Dipl.-Ing.Dr.mont. Herbert Hofstätter

I declare in lieu of oath, that I wrote this thesis and performed the associated research myself,
using only literature cited in this volume.



Monika Mörtl

June 2009

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Abstract

Is it better to consolidate an entire production zone prone to sand influx at least a little or is it preferable to consolidate a little properly?

Even in case of discovering the most challenging downhole environments for applying chemical consolidation as sand control technique this question never arises. In sand control it is not an option to prevent sand influx only from part of the productive horizons. Further it can not be tolerated to consolidate the whole production zone insufficiently and non-uniform.

Permeability anisotropy over long production intervals will favour non-uniform resin distribution which results in consolidated formation layers showing various compressive strengths. In order to achieve uniform sand consolidation over the productive zones proper resin diversion is necessary.

In the course of this thesis the suitability of three fluid loss additives regarding their diversion ability has been tested based on the following concept. The diverting agent and the resin should be pumped in the same stage of the consolidation treatment. Therefore the resin has to invade the formation as spurt loss before the fluid loss additive plugs the pore space and diverts the remaining resin to lower permeable zones.

This concept raises the need for a high spurt loss in order to achieve an appropriate consolidation radius around the wellbore. In addition to their ability to create high spurt loss and good fluid loss control over time suitable fluid loss additives have to show low susceptibility to vary spurt loss with formation permeability.

Testing showed that within a small temperature spot one of the tested products meets the prerequisites of the defined concept. The thin and uniform filter cakes created during testing showed high spurt values and good fluid loss control over time as well as low variations in spurt loss with test bed permeability.

Kurzfassung

Ist es besser einen Sand produzierenden Horizont mittels chemischer Methoden über das ganze Intervall hinweg zu verfestigen oder nur ein Stück davon technisch einwandfrei zu verfestigen?

Diese Frage darf sich nicht einmal dann stellen, wenn unter Tage extrem schwierige Bedingungen zur Sandverfestigung angetroffen werden. In der Regulierung von Sand Zufluss ist es keine Option den Zufluss aus nur einem Teil der unverfestigten Lagen zu kontrollieren. Ausserdem kann durch die ungleichmäßige Verteilung von Harz über das gesamte Intervall Sand Zufluss nicht hinreichend kontrolliert werden.

Die Anisotropie der Permeabilität einzelner Horizonte einer Lagerstätte fördert die unregelmäßige Verteilung des verpumpten Harzes und damit die Unterschiede in der Festigkeit der behandelten Lagen. Um die gleichmäßige Verteilung des Harzes über das gesamte Produktionsintervall zu gewährleisten ist die Umlenkung des Harzes in Horizonte mit niedrigerer Permeabilität notwendig.

Im Zuge dieser Arbeit wurde die Eignung dreier Zusätze zur Verhinderung von Flüssigkeitsverlusten hinsichtlich ihrer Fähigkeit zur Umverteilung des Harzstromes getestet. Die Ergebnisse wurden basierend auf folgendem Konzept analysiert. Die Zusätze zur Harzumverteilung sollen zusammen mit dem Harz verpumpt werden. Daher muss das Harz in die Formation gelangen bevor sich ein Filterkuchen an der Formation aufbauen kann und weiteren Harzfluss in die Formation verhindert und ihn in Horizonte mit geringerer Permeabilität umleitet.

Dieses Konzept ruft in den Eigenschaften des Produktes die Notwendigkeit für hohen Flüssigkeitsverlust vor dem Aufbau des Filterkuchens hervor. Dieser Anfangsverlust sollte so groß wie möglich sein um einen ausreichend großen Radius um das Bohrloch zu verfestigen. Zusätzlich zu den großen Anfangsverlusten vor Aufbau des Filterkuchens und der guten Flüssigkeitskontrolle über Zeit darf ein Produkt, das für dieses Konzept geeignet ist, diese Eigenschaften nur geringfügig mit der Permeabilität der Formation verändern. Denn nur so kann gleichmäßige Harzverteilung über ein längeres Produktionsintervall garantiert werden.

Tests zeigten, dass für eines der drei Produkte eine Temperaturnische existiert in der es genau diese Voraussetzungen erfüllt und ebenfalls vergleichsweise geringe Anfälligkeit auf Änderungen der Permeabilität zeigt.

Introduction

Unlike acid stimulation in horizontal wellbores the well known 25-75 rule is not applicable when it comes to sand consolidation. It may happen that proper acidizing of the entire well is not possible. This poses the question if it is better to acidize the entire well at least a little or if it is preferable to acidize a little properly? From several studies the 25-75 rule was derived. This rule states that if you acidize 25% of the length properly you will get 75% of the productive potential if you acidized the entire length properly. ^[13]

Sand consolidation on the other hand has to be uniform. If it is not possible to consolidate the entire interval length prone to sand production another sand control method should be chosen.

In sand consolidation a resin treatment is injected into the formation which hardens to plastic and creates artificial bonding between the loose sand grains. The reason leading to problems in distribution of the resin is permeability anisotropy over a productive interval. Treatment fluid will flow in high permeable horizons leaving the lower permeable zones untreated. This behaviour explains the need for diverting agents which allow uniform distribution of treatment fluid to all layers. Chemical diverting agents are used to build filter cakes on the formation wall and thereby equalize the permeabilities all over the interval.

The objective of this work is to test fluid loss additives for its suitability of a specific diversion concept. The concept is not based on creating about equal permeabilities all over the zone. The objective within this concept is to transport the treatment fluid in the formation by spurt loss before the fluid loss additive shuts off fluid flow to the higher permeable zones and divert the treatment fluid to other zones where again the fluid should enter the formation via spurt loss. As resin treatment requires a certain radius of consolidation this spurt loss is needed to be high and not as low as possible as often required for other treatments.

The question arises if one of the tested fluid loss products can combine extraordinary sealing abilities and high spurt losses. As the permeabilities vary across productive intervals this concept requires that lower permeability does not lead to significant decrease in spurt loss. The spurt loss defines the amount of resin in the formation and therefore the uniformity of the treatment distribution is directly connected to the spurt loss. Ideally the formation permeability would not play a role in spurt loss and would just depend on the fluid loss additive concentration added to the carrier fluid.

Filter cake build up and performance are influenced by carrier fluid composition, temperature, pressure and concentration of the fluid loss additive. The testing should be performed on sand discs as well as unconsolidated sand packs using brine as well as resin as carrier fluid for the fluid loss products.

1 Sand Production

The production of formation sand in oil and gas wells is one of the oldest problems in the petroleum industry.^[1, 2, 3, 4] Sand production can cause costly problems considering the repair or replacement of equipment. In the worst case it can lead to the complete loss of a well or can even become the reason for environmental and personal hazards due to the erosion of safety installations.^[2]

This chapter discusses the causes of sand production and the resultant consequences. Several methods to prevent sand production are described and finally an overview about necessary considerations during choosing a sand control method is given.

1.1 Definition

Within the strata of reservoir formations one have to distinguish between the load bearing part of a formation which actually supports the effective stress caused by the overburden and fine particles (fines) associated with formation fluids. The second are not part of the mechanical structure of a formation and the production of these fines prevents blocking of the flow channels and thus permeability impairment. Therefore sand control always refers to the control of load bearing formation particles.^[1, 4]

1.2 Factors influencing the Sand Production tendency of a well

The pressure difference between wellbore and reservoir allows fluid production. This fluid flow imposes stresses on the sand grains forming the mechanical structure of a reservoir. As long as these stresses do not exceed the restraining forces arising from natural cementation, reservoir pressure, capillary forces and intergranular friction sand production will not occur.^[1, 5] There are plenty of factors which can influence a wells tendency for sand production. According to Suman Jr. et al.^[6] these factors include:

- Reservoir depth and pressure
- Flow rate
- Formation cementation
- Compressibility and natural permeability as well as permeability impairment
- Surface exposed to flow (length of production interval, open vs. plugged perforations)
- Type and composition of produced fluid and amount of fluid phases
- Characteristics of formation sand as angularity or shaliness
- Pressure drawdown
- In underground gas storage the injection and withdrawal circles can lead to increasing reduction of connate water saturation and therefore to dehydration of the reservoir.

When it comes to sand production these factors should always be taken into account. In the following some important factors are described in more detail.

1.2.1 Geology

Sand production is a worldwide problem occurring in sandstone reservoirs. Geologically young (Tertiary Age) and shallow formations as well as formations in areas of tectonic stresses are prone to sand production. As a rule of thumb it can be stated that a formation having a compressive strength of more than 1000psi will not produce sand (proper completion and production methods

assumed). The compressive strength of a formation results from the intergranular bonds between the formation solids. This natural consolidation is usually much better in geologically older reservoir rocks. In case the pressure drawdown around a wellbore is favourable sand-free production is also possible for formations having a compressive strength below 1000psi. Extreme high pressure differences (from fluid withdrawal as well as injection) on the other hand can cause failure of reservoir rocks showing a higher compressive strength.^[1]

1.2.2 Fluid Production

Pressure differences, fluid frictional forces and the effective stress (weight of the overburden reduced by the reservoir pore pressure) impose stress on the load bearing sand grains of a reservoir. The major stresses responsible for rock failure and thus sand production result from flow of reservoir fluids. Fluid flow involves the creation of drag forces which increase with higher production rates and higher fluid viscosity. As fluid flow is proportional to the pressure drawdown a combination of these two mechanisms causes sand production. For many wells a maximum sand free rate or threshold production rate was observed. Below this critical rate no rock failure occurred. In order to define if the maximum sand-free rate is above the economical production rate for a certain well an economic feasibility study has to be conducted.^[1] Improper completion practices as swabbing a well too hard or bringing the well to a too high offtake capacity too quickly can cause severe changes in reservoir stress conditions and therefore be a cause of formation failure. Producing a well on maximum rate too early can lead to premature water breakthrough and weak natural cementation can be dissolved by the water although the well might have produced sand-free for quite some time. Also the misuse of acid during stimulation jobs can dissolve intergranular bonds between sand grains (for example calcium carbonate (CaCO₃) is dissolved by hydrochloric acid (HCl)).^[3]

1.2.3 Time

Many factors taken into consideration in order to predict sand production are likely to change with time. To come up with the most accurate prediction for the whole lifetime of a well these factors have to be taken into account. In this case a tectonically relaxed area is assumed which means the maximum stress is imposed by the overburden.

The sand grains as well as the reservoir fluids support the weight of the overburden. The difference between the overburden stress and the reservoir pressure gives the effective stress acting on the formation solids. As the well is on production a certain fluid volume is removed from the pore space in the reservoir and therefore the pore pressure drops. This reservoir pressure drop leads to an increase in the effective stress. The consequences can be compaction of sand grains, subsidence, and rock failure.^[1, 3, 5] The relation between overburden pressure, pore pressure and effective stress is illustrated in Figure 1.

Formation compressibility depends on in-situ loading conditions, size and shape of formation grains as well as on the mineral composition and the grain packing arrangement which is one of the factors defining the pore volume of a reservoir. Equation 1.1 gives an estimated value for the vertical compaction ΔH .

$$\Delta H = \left(\frac{\Delta e}{1 + e_0} \right) H \quad \text{or} \quad \Delta H = \left(1 - \frac{1 - \Phi_1}{1 - \Phi_2} \right) H \quad (\text{Eq. 1.1})^{[6]}$$

- H = thickness of the zone (length in [m] or [ft])
- ΔH = vertical compaction (length in [m] or [ft])
- Δe = change in void ratio (void ratio = volume of voids / volume of solids) [-]
- e_0 = original void ratio [-]
- Φ = decimal porosity *100 [%]

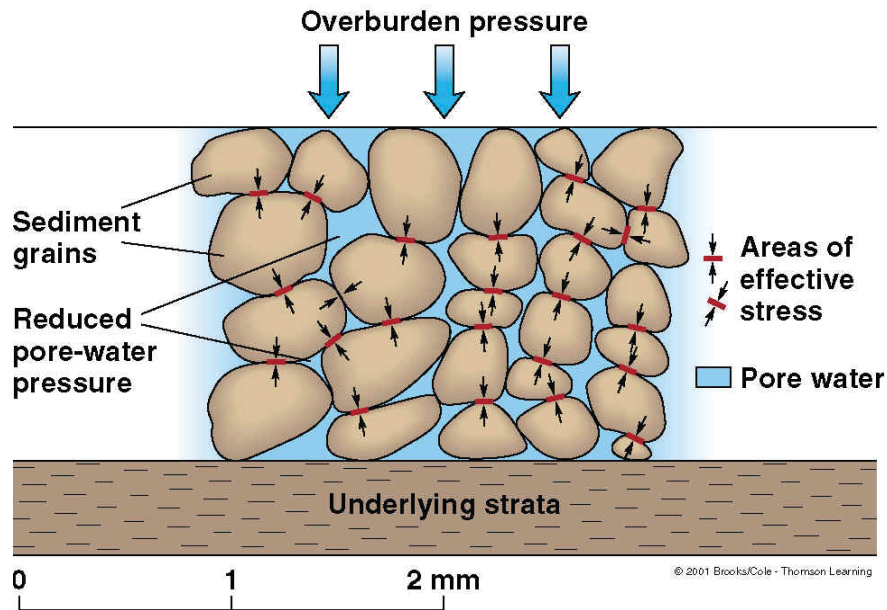


Figure 1: Relation between overburden pressure, pore pressure and effective stress [7]

Most likely the actual compaction is less than this calculated value because the formula does not take into account the structural strength added to the overburden by a sharp geologic fold and the unrelieved tectonic stresses which may be trapped in folded structures. Further inaccurate test data, the width/depth ratio of producing zones are not considered in this calculation. [6]

When starting production from an undersaturated oil reservoir (original reservoir pressure is above the bubble point pressure of the reservoir fluid) you get singlephase flow. During ongoing depletion the reservoir pressure drops below the bubble point pressure and a gas cap forms. Water breakthrough from an adjacent aquifer can lead to a third phase in the production system. In many wells multiphase flow leads to an increase in sand production. Intergranular cementation can be dissolved by water and capillary forces are reduced with increasing water saturation. By theoretically lowering the capillary pressure the interfacial tension may be decreased to an extent that the grains no longer remain in their position. As in most formations fine solids are considered to be water-wet the movement of water is able to mobilize fines and cause plugging of flow channels and therefore permeability reduction. Resulting from multiphase flow the reduced relative permeability to oil will lead to increased pressure drawdown to maintain a specified oil production rate. [1, 3]

1.2.4 Thermal Effects

Intergranular bonds can be destroyed by rising and lowering the temperature of a reservoir. Enhanced oil recovery (EOR) is a common method to increase the recovery factor of a reservoir. It is desired to leave as less residual oil in the reservoir as possible, therefore enhanced oil recovery follows natural depletion (primary oil recovery) and secondary recovery (for example water or gas injection). EOR methods as steam injection and fluid combustion alter the thermal environment of the reservoir. These thermal effects may cause sand production. Aside from EOR remedial interventions can cause thermal alteration of the reservoir. In order to get rid of gas hydrates in a production or storage reservoir one can lower the pressure or increase the temperature to melt the hydrates. Melting of hydrates would require steam injection, hot brine injection or fire flood. [1, 8]

1.3 Prediction of Sand Production

There are some techniques and guidelines used to predict the need for sand control. Unfortunately none of these techniques is applicable all over the world. Therefore a good method is to evaluate

the tendency of sand production on a reservoir-by-reservoir basis by performing extended production tests on some wells. Wells producing from the same reservoir tend to behave the same.

Mostly the rock's compressive strength is determined previous to deciding whether sand control will be needed. Ideal test result would be obtained from core samples. The compressive strength can be directly compared to the pressure drawdown. Research showed that rock will fail as soon as the drawdown pressure exceeds about 1.7 times the compressive rock strength. The Brinell hardness of rock is also used as an indicator whether sand control is required, but hardness can not be related to drawdown as easy as compressive strength.

Another guideline to determine the need for sand control is the formation porosity. As a rule of thumb it can be stated that in case the porosity is above 30% a high probability of the need for sand control exists. If the porosity is lower than 20% natural cementation is considered to be high enough to reduce the need for sand control to a minimum.

As the formation porosity is related to formation strength and sonic travel time another prediction method is logging. The sonic log records the time required for sound waves to travel through the formation, the unit used is usually microseconds. Travel times of less than 50 microseconds are an indication for a low porous, hard and dense formation. High-porosity rock shows travel times of 95 microseconds or more. Service companies offer formation property logs which predict the need for sand control by using the results of sonic, density and neutron logs. The formation property log usually overpredicts the need for sand control.

Using finite element analysis to predict the occurrence of sand production is one of the state-of-the-art methods to evaluate whether sand control should be applied or not. This method uses geomechanical numerical models in order to analyse fluid flow through the reservoir rock related to the strength of the formation.^[5] The fluid composition is not included in this analysis.

Aside from all other techniques observing offset wells and adjacent reservoir behaviour is a good approach when it comes to evaluate the need for sand control of a specific well.

1.4 Consequences resulting from Sand Production

Several operational, economical and hazardous problems can be caused by sand production. There is a wide spectrum of possible damages which reach from simple disposal and maintenance work to costly repairs and workovers as well as to complete destruction of the downhole installation and abandonment of the well. Excessive sand production can lead to formation collapse and complete loss of porosity and permeability in the formation. In the worst case serious HSE concerns can arise from sand production.^[1, 3]

1.4.1 Equipment

As mentioned before the severity of problems caused by sand production can vary a lot. In the best case scenario inconvenient and time consuming periodic sand removal from surface facilities as flowlines, manifolds and separators has to be done. While the disposal of produced formation sand is not a big problem onshore it is on offshore platforms. Due to the strict regulations only sand free of any oil contamination can be disposed. Expensive treatment facilities to remove oil from the produced sand have to be positioned at the platform where room is limited anyway.^[1, 2, 3]

Sand produced at high velocities is able to cause equipment failure by erosion. The degree of erosion depends on the fluid phase distribution, character (for example quartz may cause a higher degree of erosion than calcite) and concentration of solids, flow velocity, angle of incidence, corrosiveness of the fluids and the material (steel grade) of flow lines, screens, rod strings, valves and chokes. Equipment erosion especially occurs at or near changes in flow direction and cross section (chokes, ells).^[6] In corrosive environments sand production can lead to erosion corrosion. The erosion of metal parts and the subsequent exposure of bare metal favour corrosion. Erosion corrosion severity varies with the steel grade. In general erosion is more severe in turbulent flow regimes and in gas producing wells due to their higher flow velocity. The worst case would be erosion of downhole and surface equipment to an extent that oil or gas can escape. The

consequences of these unexpected leaks can be large economic losses resulting from production shut down and replacement of the damaged parts as well as serious safety and environmental hazards especially offshore and in environmentally sensitive areas. Subsurface safety valves can become inoperable by erosion and thus get an extreme safety hazard. ^[1, 2, 3] Facilities engineers define a flow rate of 30m/sec in order to be in a safe range regarding surface facility erosion.

Sand production can create void spaces and cause slumping of overlying formation sands. This results in abnormal loading conditions on the casing and can lead to casing deformation. This happens due to the lack of lateral support from the formation sand which is vanished by production.

^[1] Casing failure can also be caused by shear off of the casing.

1.4.2 Economics

Production loss caused by well shut-in in order to remove sand from the surface equipment or to replace worn chokes and valves can pose a reduction in hydrocarbon exploitation time and therefore lead to financial losses. Periodic sand removal from the surface equipment means the fluid velocity of the well is high enough to transport the sand grains to the surface. This means the fluid velocity exceeds the particle settling velocity given by Stokes' Law.

$$v_t = \frac{gd_p^2(\rho_p - \rho_f)}{18\mu} \quad (\text{Eq. 1.2})^{[9]}$$

v_t = particle settling velocity [m/s]

d_p = particle diameter [m]

ρ_p = particle density [kg/m³]

g = gravity constant [m/sec²]

ρ_f = fluid density [kg/m³]

μ = fluid viscosity [Pa s]

Stokes' Law is valid for spherical particles settling in Newtonian fluids it has to be modified for the use of non-Newtonian fluids. Equation 1.2 shows that the settling velocity is proportional to the particle diameter and density. The settling velocity will also increase in case the fluid density and viscosity decrease. Economic losses can also occur in case the fluid velocity is below the particle settling velocity and therefore not high enough to transport the sand grains up the production string. Subsequently the grains will accumulate downhole. At first the rathole will be filled with sand and with continued sand influx the perforated interval will be covered with sand entirely. The production rate declines and finally the well will be "sanded-up" and will need remedial operations to re-establish production. Apart from the loss of money due to the production stop the cleanout techniques involve expensive workover time. The accumulated sand can be removed by multiple wireline runs using a "bailer" or by running smaller diameter tubing or coiled tubing (velocity string) in the production string and circulate the sand out. The production loss and additional well maintenance costs are proportional to the frequency of these sand removals. In case production does not cease due to the formation of sand bridges in the production tubulars it can be proved by Darcy's Law that even a thin sand bridge can reduce the flow rate substantially and therefore the profit.

$$q = -\frac{kA dp}{\mu dL} \quad (\text{Eq. 1.3})^{[10]}$$

q = fluid flow rate through the porous medium [cm³/sec]

k = permeability [D]

A = cross-sectional area across which fluid flow occurs [cm^2]

dp/dL = pressure drop per unit length [atm/cm]

μ = fluid viscosity [cp]

The negative sign is needed because the pressure gradient is negative in the direction of flow. ^[10]
The worst case in terms of economic damage would be excessive sand production that leads to permanent reservoir damage. By producing sand from a reservoir the overlying formation will lack support and may collapse which means the sand grains will rearrange. This rearrangement will lead to a different porosity and permeability which usually are lower than originally. The reduction in porosity and permeability will be worst in case of a wide grain size distribution or high clay content within the formation interval. In case an overlying shale horizon collapses it may shut down your permeability completely and the productivity may be lost. If collapsed sand plugs the perforations the pressure drawdown in the near wellbore region increases for a defined production rate. ^[1, 3, 5, 9]

1.4.3 HSE

Serious environmental threads and hazard for personal safety can be created by equipment erosion. If erosion occurs in the long run or is very severe for a short period of time surface as well as downhole equipment can fail. In remote locations or pipe sections which are not monitored regularly the lack of performing maintenance can lead to escape of fluids. These spills can cause environmental pollution, devastating publicity and immense costs. Chokes and valves can become inoperable because of erosion. A special thread would be an inoperable subsurface safety valve (SSSV) of a gas production plant or a gas storage facility. ^[2, 5]

Apart from financial consequences in case of causing hazards for humans and environment a company may also face to loose its license to operate. People will not trust this company any longer and boycott projects in their neighbourhood. As corporate social responsibility is integrated in the policy of all major oil companies environmental sustainability is a big issue throughout their operations.

1.5 Methods to control Sand Production

In order to avoid the occurrence of above mentioned consequences of sand production several sand control methods have been developed up to now. In this section four groups of sand control methods are described apart from the passive approach as maintenance and workover.

1.5.1 Reduction of drag forces

The reduction of drag forces is considered the cheapest and often most effective way to control sand influx. Drag forces can be reduced by increasing the area open to flow and by decreasing the fluid production rate of the well. ^[4]

1.5.1.1 Increase of flow area

According to Thomas O. Allen et al. ^[4] the fluid production rate causing the movement of sand has to be considered as a rate-per-unit area of permeable formation connected to the borehole. The flow area is increased by ensuring to create clean and large perforations throughout the entire producing interval. Raise the perforation density and opening increased length of producing sections result in higher flow area. ^[4] Conventional completions are usually perforated at 8 to 12 shots per foot while tubingless completions ordinarily only have 4 shots/foot. ^[1] Another option to increase the flow area is to install a packed fracture providing a conductive path into the formation. A well designed perforation job and accurate cleaning after firing the charges are essential factors in reducing the probability of facing a serious sanding problem. Perforating debris remaining downhole can prevent uniform placement of sand control techniques. ^[4]

1.5.1.2 Production rate restriction

Drag forces causing sand production rise with increasing production rates. Therefore restricting the production rates can lead to sand-free production or decrease sand production to an acceptable level. Problems may result if the maximum sand-free rate of a well is far below the flow potential of the well and the operating company faces significant production losses resulting by restricting the flow rates.

Reducing a wells production rate to control sand production sounds pretty easy but in fact it is trial-and-error method. This is because you do not just choose any sand-free rate; the objective is to find the maximum sand-free rate under the current reservoir conditions. Due to the alteration of reservoir pressure, flow rate and water cut determination of the maximum sand-free rate has to be repeated once in a while. A method to determine the maximum sand-free rate is the “Bean-up” technique. For analysis liquid and sand rate are plotted against time. This plot shows that with each increase of production rate the sand concentration rises too and finally tapers off to the original concentration.^[4] This can be explained by the fact that unconsolidated sand grains may form a dome-shaped arch over a perforation hole. The stability of those arches depends on the packing density of the sand grains.^[6] The compressive strength of the formation, the flow rate as well as the size of the sand and the perforations influence the creation of sand arches. Sand arches are not stable and therefore can not be considered as a reliable method for sand control throughout the lifetime of a well.^[5] The surge effect of increasing the production rate breaks unstable sand bridges and increases sand production until the sand arches are reformed at the higher flow rate. Sand bridges around the perforations will reform as long as the critical flow rate is not exceeded. Once exceeded sand production continues at high rates and the reformation of sand arches is no longer possible. In order to re-establish their build up the flow rate has to be reduced significantly below the determined critical range. Once the arches are established and sand production decreases the flow rate can be increased again and adjusted somewhat below the critical rate. Using this method over a period of several months to determine the maximum sand-free rate provides ideal clean up of fines in the flow paths around the wellbore. Often higher productivity indices result from this procedure.^[4] Often workover jobs impact a wells tendency to sand production by the swab and surge pressures occurring during the jobs.

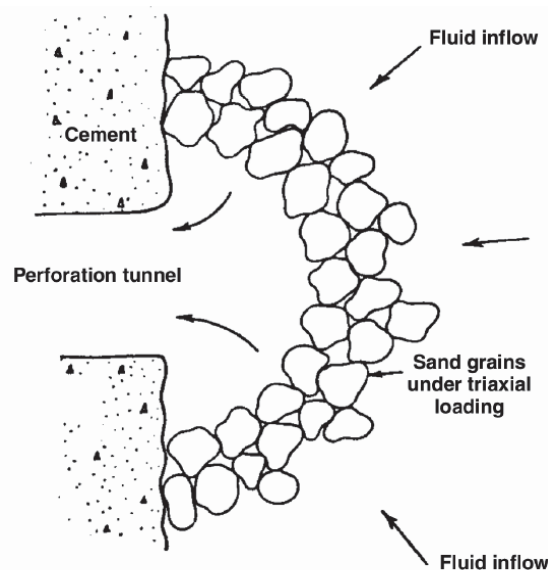


Figure 2: Stable arches building up around perforations^[5, 6]

Which rate of sand production is acceptable and which one is “excessive” has to be defined by the operating company. The limits of an acceptable sand rate are defined based on mechanical and economic factors related to probability of well sand-up and subsequent workover cost, production loss and damage of formation, casing collapse and possible hazards caused by erosion of surface and downhole equipment.^[4] In fact a risk analysis is performed to evaluate the production rate.

1.5.2 Mechanical Methods

Mechanical sand control methods involve the installation of downhole equipment that bridges off formation sand. The downhole equipment consists of gravel packs, slotted liners, wire-wrapped screens, expendables and pre-packs.

1.5.2.1 Gravel Pack

Gravel packing is the most common used sand control method. It involves installing a downhole filter to keep formation sand from entering the wellbore and causing pipe erosion. A screen or slotted liner is placed in the wellbore and packed with accurately sized gravel. The gravel pack is designed to prevent finer formation sands from entering the pack but also to be as permeable as possible.^[1]

The first challenge in designing a gravel pack is to obtain representative formation sand samples and have it analysed. A sieve analysis is performed and the formation sand recovered by each sieve is weighed and a sieve-analysis curve is constructed, where the slope of the curve represents the sand's uniformity. The opening size of the retaining sieve is plotted versus the cumulative percentage per weight retained. Highly uniform sand is represented by an almost vertical curve while a low slope indicates non-uniform sand. To calculate the so called uniformity coefficient the dimension of the grain size at the 40th percentile is divided by that at the 90th percentile $C_u = d_{40}/d_{90}$. This is used as a common basis to compare sands. Below a C_u of 3 the sand is considered uniform, if the value lies between 3 and 5 the sand is non-uniform and above 5 it is highly non-uniform sand.^[1]

The most important parameter in gravel pack design is the gravel/sand size ratio (the ratio of gravel sand size to formation sand size at equal percentile points (d_{50})). At a high gravel/sand ratio the gravel pack is invaded by formation sand and results in reduction of the permeability. Undersized gravel will give good sand control but poses the risk of reducing the productivity. As the diagram below shows the optimal gravel/sand ratio is about 5 to 6. This is where the permeability reaches its maximum and sand is still controlled. The permeability would also be good at a ratio of 15 but the formation sand would move through the gravel pack. At a ratio of 10 the formation sand will move into the gravel pack but will not be able to move through it, which results in the lowest permeability values and reduction of permeability.^[1]

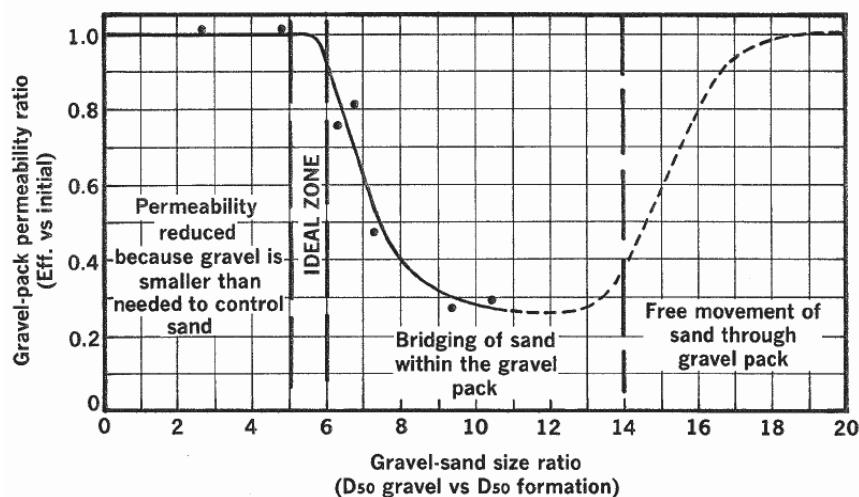


Figure 3: Effect of gravel-sand ratio on gravel pack permeability^[4]

1.5.2.2 Bare Screens, Slotted Liners and Prepacked Screens

There may be cases where installing a gravel pack may not be the best solution. As an alternative the usage of standalone screens, slotted liners or prepacked screens are options. As no gravel surrounds the screens using this sand control method the screen itself acts as a filter by bridging off

formation grains at the screen slots. In case of encountering poor quality formation sand (see chapter 1.6) the anticipated lifetime of such installations is greatly reduced by the fact that the slotted liner or wire-wrapped screen will be plugged by formation material. From laboratory tests on bridging it is known that a slot or a hole will be bridged off by particles if the slot width is less than two particle diameters. Sometimes engineers design the slot width to be equal the grain size of the 10-percentile point of the formation sands sieve analysis. As can be seen in the figure below 10% of the formation grains are now larger than the slot opening and will be stopped at the screen. Subsequently the remaining smaller sand will be stopped by the larger part of the sand grains. By changing the production rate of a well established sand bridges may break down. Further resorting of the formation sand over time may result in plugging of the slotted liner or wire-wrapped screen.^[5]

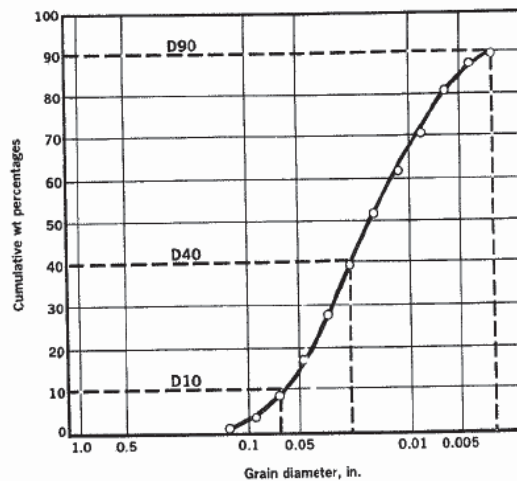


Figure 4: Sieve analysis ^[4]

As a rule of thumb standalone screens are not preferable over gravel packs in high-rate wells. ^[1] In high rate wells erosion of the screen may occur before a sand bridge can be formed to prevent further impact of sand on the bare metal. ^[5] Further it is even more unfavourable to use standalone screens in cased holes than it is in open holes. Due to the smaller flow area fluid flow through perforations will cause a concentrated impact on the screen and therefore accelerate the erosion process.

1.5.3 Chemical Methods

Plastic sand consolidation will be described in detail in chapter 2. This method involves the injection of resin into the formation pore space. The resin coats the formation grains after curing of the resin the formation grains are bonded together. The unconsolidated grains are now consolidated and therefore the formations compressive strength has been increased. ^[1] The needed chemicals for this kind of sand control treatment are expensive; therefore gravel packing is normally cheaper than sand consolidation. The length of the interval prone to sand production does influence the treatment costs of consolidation, but other than in case of setting a gravel pack the treatment can be performed after the rig has moved. ^[6]

1.5.4 Combination Methods

This type of treatment combines the technology of mechanical and chemical sand control methods.

1.5.4.1 Resin-coated Gravel pack

Unlike gravel packs the resin-coated gravel pack technique does not require a screen or liner to hold the placed gravel in position. The gravel is consolidated with resin which once fully cured adds compressive strength to the formation and prevents sand production. ^[1, 2]

To place the resin-coated gravel in the wellbore the gravel is coated with a resin and pumped downhole in slurries. For this purpose epoxy, furan or phenolic resin can be used. After selecting the appropriate gravel size for a specific wellbore the slurry can be mixed considering the perforated interval length. By mixing the gravel it has to be taken into account that the rathole as well as a portion of the casing above the uppermost perforation has to be filled with resin. For injection a conventional workstring positioned 40 to 60ft above the perforations is used. A successful job is performed by placing gravel behind every single perforation. In fact this kind of treatment is even more challenging than placing just resin behind the perforations. A prerequisite for a successful job are clean perforations which allow the resin-coated gravel to be transported behind them. So far it has been seen that resin-coated gravel packs are best applicable as remedial treatments. In case a well has already produced sand there is enough void space behind the perforations to accommodate the injected gravel. As a rule of thumb about 1 to 2 sacks of gravel per foot perforated interval is needed to control the sand production properly. Applying resin-coated gravel packs as an initial treatment has a good chance to fail because rarely 1 sack gravel/ft can be injected during this stage of well life. Therefore it is recommended to produce the well for a short period of time in order to increase the likelihood of the formation accepting one sack of gravel per foot of perforated interval. Some experts believe that injecting the resin-coated gravel at fracturing pressure will force the gravel through more perforations and therefore improve coverage. Other experts have a controversial opinion; they are convinced fracturing causes large amounts of gravel to be conducted away from the well and insufficient gravel is left behind the perforation. In order to increase the consolidated area around the wellbore extra resin is either added to the resin gravel slurry or pumped downhole after the resin is in place. The objective of investing in this additional amount of resin is to consolidate gravel and the formation sand.^[1]

There are two options to cure the resin: It may be internally catalysed which means the catalyst is added to the resin before coating the gravel or it may be in-situ-catalysed which means that the catalyst is pumped in a separate stage after the gravel has been placed behind the perforations. Once the gravel is in place the so called internally catalysed resin is cured while the gravel is still in the casing and has to be drilled out afterwards. In-situ-catalysed resin systems can be reverse-circulated out of the borehole and usually the gravel packing procedure is repeated. The catalyst is finally injected after performing a washout.^[1]

There is the option to apply either a liquid or a solid resin coating to the gravel. The first one is produced by mixing gravel with the carrier fluid. At this point a silane coupling agent can be added to the created slurry in order to favour wetting and resin adhesion. Afterwards the liquid resin is added to the slurry to form a coating on the gravel. Methods for solid resin coating require processing equipment and therefore the resin is applied by the manufacturer before shipment to the well location. The solid resin coating may be applied by either melting the resin, mixing it with gravel and cool it down or the resin can be dissolved in a solvent, mixed with the gravel and let the solvent evaporate to leave the sand coated.^[1]

1.6 Selection of the appropriate Sand control Method

When it comes to choose the appropriate method to control formation sand influx a broad selection of techniques is available (see chapter 1.5). In the following some considerations are summarized which should be taken into account when dealing with the need for a sand control installation.

According to Suman et al.^[6] the evaluation of selection and design of sand control techniques is performed based on the following factors.

- Initial and repair cost of installation (the mean time between failure (MTBF) is a criteria by evaluation of costs)
- Reliability
- Effect on productivity
- Quality of formation sand – is defined by the nonsilica content and grain size distribution of sand. Good-quality sand shows a high permeability grace to its narrow

grain-size distribution and low nonsilica content (5-15%). Poor-quality sand has a wide particle size distribution, silica content up to 50% and low permeability. [1]

- Presence of multiple, thin pay zone horizons
- Exclusion of interbedded water or gas
- Presence of undesirable shale streaks
- Level of reservoir pressure depletion
- Sand production history

Figure 1.3 shows a schematic diagram of some treatment options available.

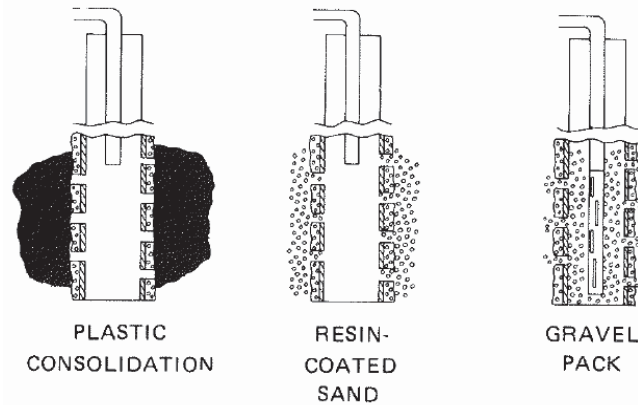


Figure 5: Sand control techniques [1]

The content of the following table is taken from Michael J. Economides et al.: “Petroleum Well Construction” [2]. It summarizes the limitations and merits of various sand control methods.

Formation characteristics	Gravel Packs and Screens	Consolidation with resins and resin-coated sands
Formation Strength	No change	Adds formation strength, with exception of the resin-coated sands
Permeability	Certain techniques may reduce permeability	Certain techniques may reduce permeability
Poorly sorted grain sizing	Applicable using special job design	Applicable with few restrictions
< 10% Fines and Clays	Very applicable. Good anticipated job life.	Very applicable. Good anticipated job life.
> 10% Fines and Clays	Applicable using special job techniques	Marginally applicable. Good resin injection and coverage is difficult.
>10% Acid Solubility	Applicable with restricted acid pretreatments.	Not applicable with acid-hardened type resin.
<10% Acid Solubility	Very applicable. Good anticipated job life.	Very applicable. Good anticipated job life.
<50° Hole Angle	Very applicable. Good anticipated job life.	Very applicable. Good anticipated job life.
>50° Hole Angle	Applicable using special tools, screens and techniques.	Not applicable. Poor job success history, uniform coverage problems.
Open Hole	Applicable using special job techniques.	Not applicable. Poor job success history, uniform coverage problems.
Cased Hole	Very applicable. Good anticipated job life.	Usually very applicable. Good anticipated job life.
Slim Casing	Marginally applicable. Severe tool and screen restrictions.	Very applicable. Good anticipated job life.
Single Zone	Very applicable. Good anticipated job life.	Very applicable. Good anticipated job life.
Multiple Zones	Applicable. Requires special tools, screens and techniques, leaving screen in wellbore.	Very applicable. Leaves clear wellbore, should be done as an initial measure.
<30ft Interval Length	Very applicable. Good anticipated job life.	Mostly very applicable. Good anticipated job life.
>30ft Interval Length	Very applicable. Special tools, screens and	Not applicable. High costs and uneven

	designs may be required.	resin coverage.
High water producer	Applicable. Additional chemical fines control may be required.	Very applicable. Good anticipated job life.
Gas producer	Very applicable. Good anticipated job life.	Applicable. Some resin systems clean up better with good anticipated job life.
Oil producer	Very applicable. Job life depends on quality of screen and proppant used.	Very applicable. Anticipated job life of 3 to 8 years.
Low bottom hole temperature, <120°F	Very applicable. Good anticipated job life.	Provisional. Difficult curing conditions for some resins.
Medium bottom hole temperature	Very applicable. Good anticipated job life.	Very applicable. Good anticipated job life.
High bottom hole temperature, >250°F	Provisional. Special proppant and tool design may be required.	Marginally applicable. Some resins are more resistant than others.
Steam Injection	Provisional. Likely will require special proppant and tool designs.	Marginally applicable. Some resins are more resistant than others.

Table 1: Limitations and merits of sand control methods ^[2]

1.6.1.1 Gravel Pack

By variation of the screen length and opening size a gravel pack can be designed to meet the needs for controlling different formation grain sizes and interval lengths. This flexibility allows covering interval lengths of 20-200ft, but also treatments covering 500ft are reported. As a drilling rig or a coiled tubing unit is required to perform a gravel pack job, the initial cost of this sand control method is directly proportional to the rig costs. While moderate costs arise from a job performed by a land rig a gravel pack job may become very expensive offshore. Although a gravel pack can be designed for various wellbore requirements it is not without disadvantages. In case the screen positioned downhole fails its replacement require a fishing job which generates additional cost. It may occur that the use of a special gravel-packing fluid is required. This may happen in overpressured formations where calcium chloride brine can not be used to control the formation pressure (in this case it is possible to use potassium carbonate in order to obtain higher completion fluid densities). Further formation fines may invade the gravel pack, plug the pore throats and create a severe impairment of productivity.

Another factor that should be considered during the selection of sand control techniques is the wellbore geometry. While gravel packs are very successful in conventional completions tubingless completions lower the success rate of gravel packs. The screen positioned in a tubingless completion has a smaller diameter and a small slot area which is more prone to plugging by fines. Further due to the small wellbore diameter less than 1inch of gravel is placed between the screen and the perforations. Even if the screen could be placed perfectly centralized the chances are great that formation sand reaches the screen and causes failure of the installation. Also tubingless wells are usually perforated at 4 shots per foot. The result is reduced flow area compared to the perforation at 8 to 12 shots/ft.

Regarding the well trajectory it could be determined that for wells with an inclination above 45° proper placement designs are required to succeed in totally filling the annulus with gravel.

Sand quality is directly proportional to the success of all sand control methods. Ordinarily gravel packs are too coarse to control fines of poor-quality formation sand. Once invaded the pack the fines will plug the screen or cause its erosion and subsequent failure. When facing the risk of erosion it is important to take into account the type of reservoir fluid encountered during production. Turbulent gas streams transporting formation fines are a special thread to mechanical equipment.

Basically gravel packs can be applied widely independent of reservoir temperature. Special completion fluids may be required in case of installing a sand control technique in an abnormally pressured zone. ^[1]

1.6.1.2 Consolidation

Unlike gravel pack no mechanical parts are left in the wellbore after performing sand consolidation treatment. This makes workovers during the lifetime of a well simpler and fishing jobs can be

avoided. After consolidating the surrounding of the wellbore fines migration will not lead to productivity decline. Further as long as the well has not sanded up before considering the consolidation treatment it can be performed without a rig.

There are some significant disadvantages of sand consolidation economical as well as operational. The costs per foot for consolidation of a perforated interval are high and can become too high to be economical in long intervals (about 50ft). To make this kind of treatment work the consolidation has to be uniform therefore the treatment fluid has to be placed outside all perforations. A coverage problem can occur if one zone does not receive enough resin to consolidate the formation sand properly. Even sand production from a single perforation may cause equipment failure by erosion. The longer the perforation interval the greater the change to encounter permeability anisotropy and facing the fact that one zone will accept more treatment fluid than another.

As in case of gravel packing tubingless completion also reduces the success of plastic consolidation. In multiple tubingless wells it is difficult to accomplish a good primary cement job; channels in the cement may prevent uniform sand consolidation. So far no indications of well deviation influencing the consolidation process and success have been found.

Deteriorating sand quality leads to declining consolidation success. The chemicals used to consolidate the sand and therefore add compressive strength to the rock exhibit poorer wetting and adhesion performance with nonsilica material. Compared to gravel packs sand consolidation declines more rapid with deteriorating quality of formation sand. Further low formation permeability makes it more difficult to inject the chemicals.

The application of consolidation treatment requires the consideration of reservoir temperature. Basically all techniques can be used from 120° to 200°F. At too low temperatures the curing of the resin would take too long. At extremely high temperatures on the other hand softening of bonding agents can cause strength loss of the consolidated sand. Special resin formulas, accelerators and catalysts can extend their range of usage above and below the given temperature range. ^[1]

Suman et al. ^[6] summarizes the downhole conditions under which it may be cheaper to perform sand consolidation instead of gravel packing (taking already into account that no rig is needed).

- short treatment interval (above 10ft special placement tools are needed)
- no previous sand production (cavities formed in previous production makes uniform resin placement more difficult – prepacking)
- zones with limited sand-producing tendency
- high reservoir pressure
- sand quality has to be good and formation should have sufficient vertical permeability
- upper zones of multiple completions – no mechanical parts left in wellbore

1.6.1.3 Resin-Coated gravel

Only a limited amount of sand is consolidated in this procedure therefore the cost is reduced by the need of only 1 to 2 gal/ft resin of perforated interval. Compared to a conventional consolidation treatment the cost reduction can be between 25 to 50%. Although the cost reduction makes this method favourable for longer intervals the coverage is more severe to obtain than with conventional consolidation. ^[1]

2 Sand Consolidation

Unlike acid treatments in horizontal wellbores the well known 25-75 rule is not applicable when it comes to sand consolidation. It may happen that proper acidizing of the entire well is not possible. This poses the question if it is better to acidize the entire well at least a little or if it is preferable to acidize a little properly? From several studies the 25-75 rule was derived. This rule states that if you acidize 25% of the length properly you will get 75% of the productive potential if you acidized the entire length properly. ^[13]

Sand consolidation on the other hand has to be uniform. If it is not possible to consolidate the entire interval length prone to sand production another sand control method should be chosen. In case the entire interval is not properly consolidated the consequences resulting from sand production discussed in chapter one may occur. Therefore the need for uniform diversion of the consolidation chemicals becomes clear.

This chapter discusses the principles of plastic sand consolidation and deals with the problem of proper diversion of the resin system pumped downhole.

2.1 Principles of plastic consolidation

Within this chapter an overview on the objectives of sand consolidation is given. The purpose of some consolidation chemicals are describes as well as the actual methods used for coating the formation sand grains.

2.1.1 Objectives of sand consolidation

Unconsolidated formations lack natural grain cementation and bonding. Plastic sand consolidation is a sand control method designed to establish this lacking cementation artificially. Performing this consolidation of producing intervals properly involves technically challenging treatment design and field procedures. The objective of this treatment is in situ grain-to-grain cementation that withstands forces created during fluid production. Achieving this major objective is combined of succeeding in the following steps. It is necessary to coat the sand grains with resin and to concentrate the resin at the grain contact points for bonding them. Further the least possible reduction in permeability should be achieved therefore the major part of the pore space has to be open for flow after the resin is cured. Figure 6 illustrates these objectives. When two phases are present in porous formations the wetting phase always concentrates at the grain contact points because interfacial tension (IFT) between the wetting phase and the fluid filling the remaining pore space will draw the wetting phase in the grain contact region. This leads to the conclusion that in order to achieve proper consolidation the resin has to become the wetting phase in the formation. The resin has to be partially displaced with a non-reactive fluid before it hardens and plugs the flow channel. As a displacing fluid mostly oil is used but for some systems it can also be water. The percentage of pore volume filled with resin is about 35% but variation of this number can be caused by the consolidation systems used and the initial formation properties as permeability and porosity. ^[1, 6]

It has been mentioned before that in case of good completion and production practices a formation with a compressive strength above 1000psi will most likely not produce sand. Therefore the objective of plastic consolidation is to create a compressive strength above 1000psi. How large has the consolidated radius around a wellbore have to be in order to reach this strength level after consolidation? Assuming ideal conditions only a few inches would be enough, but as an actual downhole situation does not follow ideal conditions some factors has to be taken into account and the consolidation radius has to be adjusted. Within one productive interval horizons of several permeabilities may be encountered. These variations in formation permeability lead to a non-uniform distribution of resin because the fluids are accepted at different ratios. Further depending on the quality of the perforation job it might occur that more or less of the perforations are plugged by debris. In case of plugged perforations the vertical formation permeability plays a large role when it comes to uniform resin distribution. Unconsolidated sand behind plugged perforations has a good

chance to be treated by resin if the vertical formation permeability is good. Unfortunately in many formations so called shale breaks are encountered. The mentioned shale breaks limit vertical flow behind the casing and therefore it might be impossible to treat sand behind a plugged formation with the resin injected in adjacent perforations. As it has already been emphasized in the introduction to this chapter the consolidation of the entire interval is of major importance for a successful sand control treatment. In addition to these factors which prevent to plan for ideal consolidation conditions there is also the fact of consolidation strength loss over time that has to be taken into account. This expected strength degradation leads to an increase of consolidated radius around the borehole. From experience it has been concluded that an average consolidated radius of 2-3ft will result in proper sand control and reasonable costs. ^[1]

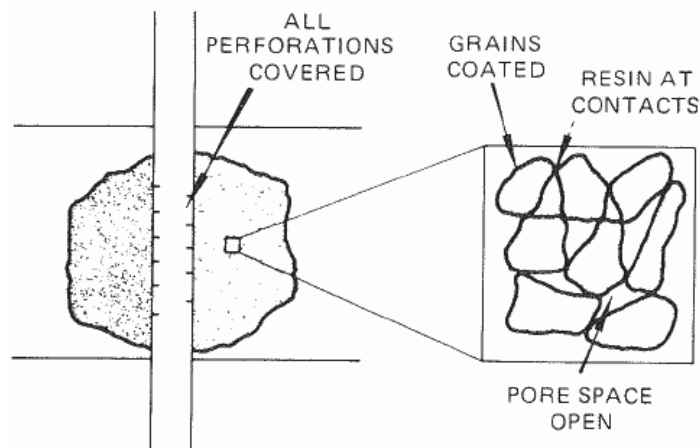


Figure 6: Objectives of plastic consolidation ^[1]

2.1.2 Chemicals used for the treatment

The chemicals needed for sand consolidation can be classified in five categories according to their function within the treatment process. There are the chemicals used for preflush, the actual resin, diluents, coupling agent and fluids used to perform an overflush after the resin is in place. ^[1]

2.1.2.1 Preflushes

In order to provide strong bonds between the sand grains the sand surface has to be wetted by the resin. Preflushes are chemicals used to ensure sufficient resin coating of the sand grains by removing natural formation fluids (crude oil or brine) which may contain constituents incompatible with the resin system. The problems can be the absorption of part of the resin by crude oils which decreased the cured resin strength and also too high or too low saline formation brine can interfere with the resin. The addition of surfactants to the preflush will lead to a more complete removal of formation fluids and favours wettability of sand by resin. Apart from oil or water preflushes also mutual solvents can be used. These products can remove both brine and oil from the pore space. As in most sand water is the wetting phase most resins perform better after removing all water from the pore space. ^[1]

2.1.2.2 Resins

The most common resin consolidation processes are based on the polymerization of an epoxy, a furan or a phenolic resin. Polymerization can proceed through catalysis or curing. The first polymerization refers to the fact that a catalyst causes the resin molecules to form long chains. Curing means that a curing agent acts directly with the resin and becomes part of the resin structure. Table 2 summarizes the properties of epoxy, furan and phenolic resins. It can be seen that no reaction byproduct is produced by epoxy resins and the resin only shrinks a little while curing. It can be stated that epoxy coatings are highly resistant to chemical attack and uniform. A loss in consolidation strength will occur at a formation temperature of 250°F. In below table it is

stated that epoxy resins do not wet sands in presence of water which means that the residual water has to be removed by a preflush. ^[1] But there are also epoxy resins available which are water based. In the experimental part of this work a water based resin system is tested on brine saturated sand packs. In order to consolidate the sand it was not necessary to remove the brine by with a preflush.

Resin	Catalyzed or Cured by	Reaction Byproducts	Softening Temperature (°F)	Wets Sand in Presence of Water
Epoxy	Amines	None	250	No
Furan	Strong acid	H ₂ O	300	Yes
Phenolic	Strong base	H ₂ O	300	Yes

Table 2: Summary of resin properties ^[1]

In case of using furan for consolidation the process is based on the polymerization of furfuryl alcohol by an acid catalyst. A methylene bridge is formed by furfuryl alcohol molecules. This process leads to the release of water as a reaction byproduct. An advantage of furan resin is its good resistance to chemicals, except oxidizing agents as chlorine. Another advantage lies in its ability to coat sand grains in the presence of water and the temperature limit is higher than those of the epoxy resin. Furan polymerization occur very quickly which can limit the shelf life of some resins; further the reaction byproduct collects as tiny bubbles in the coating and creates stress concentrations. Phenolic resins form a highly cross linked structure, the reaction between a phenol and an aldehyde results in a methylene bridge linking two phenol molecules. Either an acid or a base can be used as catalyst. In Table 2 it can be seen that the properties of the phenolic resins are similar to those of furans. ^[1]

2.1.2.3 Diluents

Undiluted resins have viscosities of 100 to 10000cp. It is required to dilute the resins in order to achieve a pumpable resin system. To come up with a 10-20cp diluted resin viscosity diluent levels of 10 to 50 wt% are needed. Reactive diluents are able to polymerize with resins while non-reactive diluents will not become part of the resin structure. ^[1]

2.1.2.4 Coupling Agents

Coupling agents are included in the resin formulation or added to the preflush. The second method seems to be more effective. Organosilanes are the most widely used coupling agents. During polymerization the organic end group reacts with the resin and the silane and group reacts with the sand. These reactions lead to the chemical bonding of resin to the silica surface of the sand grains. ^[1]

2.1.2.5 Overflush Fluids

After the resin has been successfully injected into the formation often an overflush is pumped. This overflush should guarantee that the sand is still permeable after consolidation by opening a flow channel through the resin. In some treatments the overflush is used to transport the catalyst into the formation to start the polymerization process. Another reason for the application of an overflush is to extract the diluent from the resin. For displacement of excess resin the overflush has to be more viscous than the resin. ^[1]

2.1.3 Methods used for sand coating

The objective of the resin treatment is to have the resin drawn to the grain –to-grain contact points and get a thin resin coating on the grains. There are two different processes to achieve these results. One process is the so called phase separation process. The diluted resin solution in a solvent is combined with an activator (a curing agent or a catalyst) and the liquid resin phase separates from the solvent after a certain period of time and is drawn to the grain-to-grain contact

points. The other process is called the overflush process which uses a high-yield resin solution. Permeability is regained not by phase separation as before but by pumping an overflush fluid in the rock and displacing the excess resin from the flow path. What remains in the pore space is residual resin saturation that wets the sand grains. The overflush is designed to control the thickness of the resin coating on the sand grains which is directly linked to control of regained permeability and compressive strength of the consolidated formation. [6]

In the following three consolidation processes are described. The names of the coating method describe the mechanism of mixing catalyst and resin (first part of the name) and also refer to the mechanism of flow path reestablishment (second part of the name). [1]

The internally catalysed phase separation describes a treatment method where the resin components and they are already mixed together before they reach the formation. The polymerized plastic precipitates from the resin solution by phase separation. The polymerization process results in two phases in the formation: plastic coating on the sand grains and solvent filling the pore space to guarantee a regain of permeability. In Figure 7a the internally catalysed process is illustrated. Before starting the consolidation treatment crude oil and formation brine fill the pore space (1). The next step (2) is a preflush which removes crude oil and residual water saturation subsequently the resin solution is injected (3). After the injection the curing of the resin starts the resin polymerizes and precipitates onto the sand (4). The carrier solvent remains in the pore space and the plastic forms on the sand grains to bond them together. The solvent is displaced when oil production starts (5). An advantage of this method is that the resin/catalyst ratio can be adjusted precisely but it has to be kept in mind that the polymerization starts as soon as the catalyst is added to the resin. Therefore the resin placement has to be finished before the phase separation starts. [1]

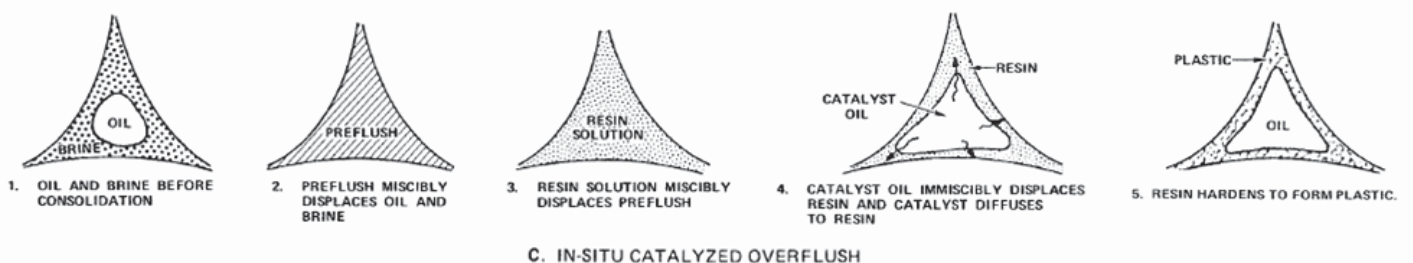
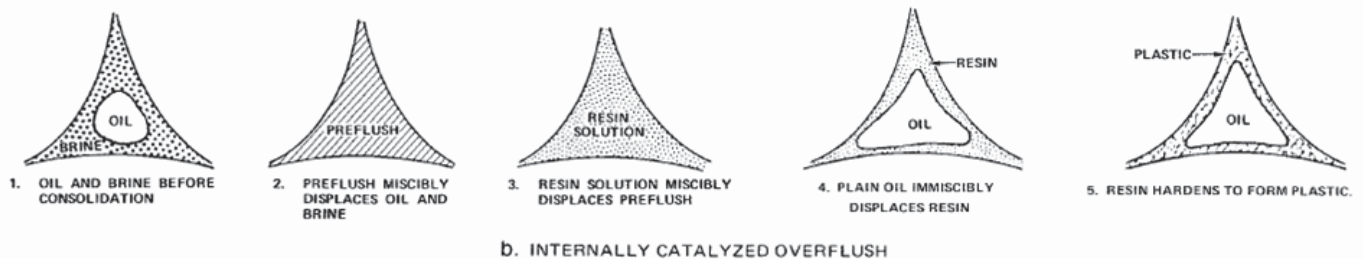
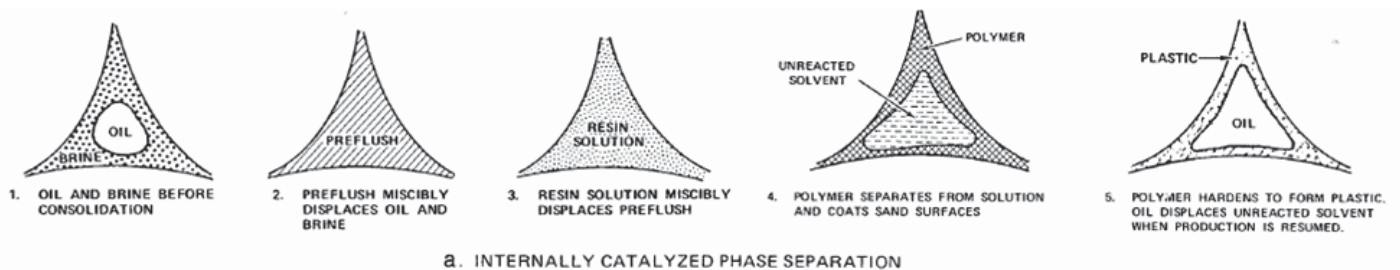


Figure 7: Sand coating methods [1]

Figure 7b shows the internally catalysed overflush process; here the resin and the hardener (catalyst) are mixed together before the treatment starts and permeability is restored by the displacement of the excess resin. Schematic (1) shows the initial downhole situation and in (2) the preflush has already been pumped in the formation. The preflush is displaced by the resin solution (3). Then an inert fluid, usually oil is used, is pumped to displace the excess resin, which means all

resin but the residual resin wetting the sand grain surfaces (4). The remaining resin saturation (30-35% of the pore volume) hardens and forms plastic cementing together the loose sand grains (5).^[1]

The in-situ catalysed overflush is displayed in Figure 7c. The first three steps are the same than in the processes described before. The difference here is that the hardener is not included when the resin is pumped in place. This means in step (3) no catalyst is in the formation and the resin would contain its initial state if no further treatment steps would follow. The catalyst is dissolved in the overflush fluid and diffuses into the resin phase when arriving in the pore space (4). With the overflush the permeability has been re-established and the resin hardens to plastic (5). This process has the advantage that pumping time is not limited; in case of a mechanical breakdown the resin could not start hardening in the tubular.^[1]

2.2 Diversion

It has already been pointed out that only uniform resin distribution over the entire production interval is a cure for sand production. Basically in long production zones various layers with different permeabilities can be encountered. Highest formation permeability poses the least resistance to fluid flow and therefore the main part of an injection treatment volume will be transported there and this leaves lower permeable formation treated inadequately. In case of sand control leaving a horizon treated inadequately will not cure the problem and sand inflow will continue resulting in damage of downhole equipment or in the worst case scenario even in hazards for human and environment. Using diversion it is possible to either focus treatment on certain zones (lowest permeable zone in acid treatments) or to distribute the treatment fluid uniformly all over the productive interval. As diversion involves the prevention or reduction of fluid flow in certain zones it is necessary that the diversion effect is only temporary and full permeability can be re-established after completing the treatment. Diversion can be obtained by using mechanical or chemical diversion methods.^[18]

Mechanical methods include ball sealers, straddle-packers, injection-type packers or solid-particle diverting agents. Ball sealers are dropped during the treatment; also solid-particle diverting agents are incorporated in the fluid system and plug the zones taking the most fluid. Injection-type packers will isolate an interval before the formation treatment starts. When using packers and straddle packer assemblies several short treatments over a longer interval are performed to guarantee evenly distribution of the treatment fluid.^[2, 18]

Chemical diverters are solid slugs, viscous pill or foams. The used solids include oil soluble resins, benzoic acid flakes, rock salt or calcium carbonate. The solids are held in suspension in a viscous carrier fluid. Viscous pills contain gelling agent with delayed cross linkers.^[2]

Mechanical diversion methods are not suitable or practical for certain applications. The methods require are applicable only in cased holes and further a perfect isolation behind the casing is required. Mechanical methods divert the flow completely from one zone the others. The chemical diversion is just based on the fact that particles will create a filter cake at higher injectivity layers and therefore divert the flow to other zones. Other than the mechanical methods the chemical products try to equalize the flow of the treating fluid per unit of thickness. In order to obtain effective flow redistribution the created filter cakes have to seal very well. Another prerequisite is that the filter cakes are easy to remove in order to prevent damage in permeability.^[19]

In chapter 3 the testing of three diverting agents is described. It should be analysed if the products are suitable to divert resin in order to get a uniform resin distribution and therefore uniform compressive strength over the entire production interval.

2.3 Fluid loss additives

Fluid loss control is a big issue in drilling as well as in completion. In drilling fluid loss additives prevent the loss of drilling mud and therefore are important for well control. Beside the safety factor which is also true during completion and workover jobs, fluid loss additives prevent the loss of highly expensive treatment fluids as resins, acids, high-density brines, drilling muds or cements.

Fluid loss control materials include foam, oil-soluble resins, acid-soluble particulates, graded salt slurries, linear viscoelastic polymers and heavy metal-crosslinked polymers.^[20]

In the practical part of this work various fluid loss materials will be tested. One product is the bridging agent Baracarb® which is calcium carbonate. One product is a Polyacrylamide gel chopped up for usage as bridging agent and the other one is borate cross linked guar.

To guarantee a quick built up of fluid loss control with bridging material it is necessary to use a wide particle size distribution. A filter cake build up starts with large particles which form the basic structure and start stopping smaller particles which plug the remaining void spaces on the basic structure. As the filter cake becomes thicker also particles at colloid size are trapped in the filter cake. Subsequently the fluid loss is decreased to a minimum. In order to achieve this seal the bridging particle size distribution has to be well designed in accordance with the formation pore sizes. The median bridging particle size should be equally or slightly larger than 1/3 median pore size of the formation. This should be true for five volume percent of all solids which should have a wide size distribution. Widely used bridging materials are oil-soluble resins, graded rock salt and calcium carbonate. Calcium carbonate is soluble in acid and oil-soluble resin can be removed by production of crude or gas condensates. The advantages of these materials are their blocky granular shape, their availability of all size distributions needed for a variety of pore throat sizes. Further the fact that particle size distribution can be controlled by manufacturing and their solubility properties.^[6]

Bridging agents as Baracarb build a filter cake by creation of a dense pack of fluid loss control material. Other fluid loss materials as the later tested FDP-S875 create seal by formation of gels. In the following some brief information on gels and gums is given. The product FDP-S875 will be described in chapter 3.

A gel is a cross-linked polymer network swollen in a liquid medium.^[21] The properties of a gel depend on the interaction of polymer network and liquid. The polymer network retains the liquid by providing a structural framework to keep it in place and the liquid prevents the polymer network from collapsing into a compact mass. Characterization of gels is done according to their equilibrium, dynamic and kinetic properties. These properties depend on the gel state which is defined by osmotic pressure, temperature, solvent composition and degree of swelling. Gels can take the following phases: sol, gaslike and liquidlike. Gel-sol transitions have already been studied but gel-gel phase transitions are not yet fully understood. The first ones affect the gel structure and properties. In a typical condensation polymerization the connection of bifunctional units leads to the formation of long chain molecules, within these chains polyfunctional units serve as cross links. Another possibility is the formation of polymer networks by cross-linking polymer chains (already formed from bifunctional units). It has to be mentioned that some gels are cross-linked chemically and others are cross-linked physically. For example acrylamide gels which are used in further testing are chemically cross-linked gels. It has been discovered that small changes of external conditions can lead to significant changes in the state of gels. By lowering the temperature polymer networks lose their elasticity and their compressibility increases. Depending on temperature a gel can swell or shrink by a factor 500. Gel transitions can be induced by alternation of the solvent composition, pH or ionic composition.^[21]

Gels can be formed out of gums. Gums are polymeric substances which form highly viscous dispersions or gels if put in an appropriate solvent or swelling agent. Gums can be natural polymers (polysaccharides, proteins and natural rubber) or chemically modified natural polymers (hydroxyethylcellulose) and synthetic polymers (poly vinyl alcohol and polyacrylamide). Industrial gums refer to polysaccharides and their derivatives and they are classified by source and structure. Within the class seed gums the following can be found: corn starch, locust bean gum and guar gum. As FDP-S875 is made from guar flour the guar will be described in the following. Commercial guar gum is the ground endosperm of guar seeds and consists of 80-85% guaran (the purified polysaccharide from guar gum), 10-14% moisture, 3-5% protein, 1-2% fiber, 0.5-1% ash and 0.4-1% lipid. The guar gum grows in semiarid regions and gets a height of 1-2m. An attrition mill splits the seeds in half, the seed halves (endosperms with adhering hull) are heated and a mill separates the hull from the endosperm which is softened by heating. The resultant "split" from this process is pure endosperm which can be converted to a high purity split by abrasion. Further these splits are often reduced to granules and powders. Guar endosperm can be derivatized. Derivatives are

produced in order to control hydration rate, peak viscosity, ash content, insoluble material, heat stability and compatibility with other materials. Fine guar granulations are rapidly hydrated. Derivatized guar as hydroxypropylguar gum (HPG) are more stable at elevated temperatures and develop viscosity more rapidly at low concentrations. The most rapid hydration can be observed at pH 6-9. Guar polymer chains can be cross-linked with borate, titanates, zirconates and antimonates. For a given solution concentration guar allows almost constant viscosity over the range of pH 3-10. The ability of gum to hydrate is decreased at a pH above 11 therefore lower viscosity is the result. The optimum hydration range is between pH 5-8. ^[21,22]



Figure 8: From guar beans to guar flour

3 Testing of fluid loss products as diverting agents in an HPHT cell

All measurements and testing of the experimental part of this thesis was conducted at the Halliburton Technology Center in Duncan, Oklahoma.

The usability of several fluid loss products as diverting agents for resin treatments will be tested within this thesis. This chapter describes the concept on which the subsequent tests are based as well as data evaluation, equipment and testing procedures. Further it discusses the performance of three fluid loss products based on the present concept.

3.1 Concept

As discussed in the previous chapter the need for resin diversion arises from permeability anisotropy in reservoir formations. With increasing length of a perforated interval, the possibility to encounter productive layers having various permeabilities increases. Figure 9 shows an interval perforating three productive horizons with different permeabilities. The given formation is prone to sand production and it has been decided to consolidate the near wellbore area with resin. The right hand side of the sketch below illustrates the distribution of the injected resin treatment if no diversion measures are taken. The largest portion of resin (in the sketch shown yellow) would be injected in the horizon showing the highest permeability (1000mD). In this example the other layers would receive only a small fraction of resin resulting in non-uniform resin distribution over the perforated length. Therefore sand production might not be prevented from all formation layers. Ongoing sand production may occur because the non-uniform resin distribution results in various compressive strengths of the reservoir layers and some of them may be too weak to withstand drag forces occurring during fluid production and forces resulting from surge and swab pressures. On the left side of the sketch ideal uniform resin distribution over the whole interval length is shown. Here the resin is diverted to the lower permeable formations by plugging the flow paths into the high permeability horizon.

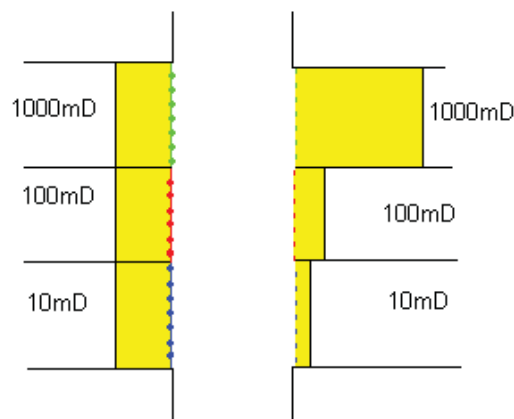


Figure 9: Simplified and idealized sketch of diversion mechanism (not true to scale)

As mentioned in the previous chapter there are several methods to divert all kinds of formation treatments as for example acids or resins. The tests performed in the course of this thesis are based on the following diversion concept.

The diverting agent and the resin should be pumped in the same stage of the consolidation treatment. The entire amount of resin needed for consolidation should invade the formation as spurt loss (leak off occurring while the fluid-retaining wall is being built up^[12]) before the fluid loss product plugs the pore space and diverts parts of the resin to other productive horizons.

Within this work the term spurt loss is referred to as fluid leak off (gal) with respect to surface area (ft^2) at a defined pressure difference Δp . Therefore the calculated spurt loss values are given in gal/ft^2 .

The suitability of three fluid loss additives will be tested based on this concept. In this case it is not wanted that after a small spurt loss a filter cake is deposited on the formation and results in a low permeability diverting the resin to other zones. On the other zones filter cakes with about the same permeability are created and this procedure continues until the entire productive zone has about equal permeability. This would also lead to resin diversion but by generating about equal permeability over the production interval. The objective of this project is to find a product that has spurt values as high as possible and a filter cake permeability as low as possible. With these properties the resin penetration depth completely depends on spurt loss. Therefore the products abilities to create high spurt values should be independent of formation permeability.

This concept raises the need for a high spurt loss in order to achieve an appropriate consolidation radius around the wellbore. Apart from that this one stage treatment requires that although permeability varies across productive zones the spurt loss remains approximately the same. Although this may not be realistic for extreme high permeability anisotropy it might be true for less severe alterations in permeability. The testing will show if a permeability range can be defined within which this concept is applicable.

In an openhole completion the cross sectional area across which flow occurs is the surface area of a cylinder given by $2\pi r h$. The resin volume leaking off into the formation is given by the volume of the annulus ($[R^2 - r^2] \pi h$) times the porosity of the formation (Figure 10 and 11).

Using these equations and dividing the resin volume ($[R^2 - r^2] \pi h \Phi$) by the wellbore surface area ($2\pi r h$) the fluid leak off per unit wellbore surface area can be calculated. This concept neglects the connate water saturation of a formation which reduces the resin volume by about 30%.

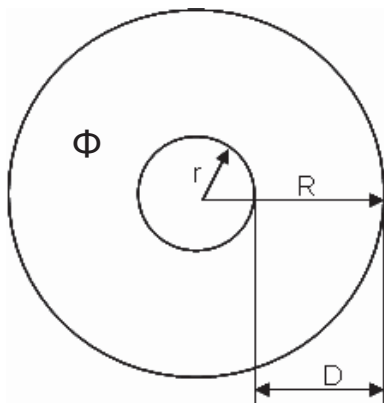


Figure 10: Resin penetration depth top view

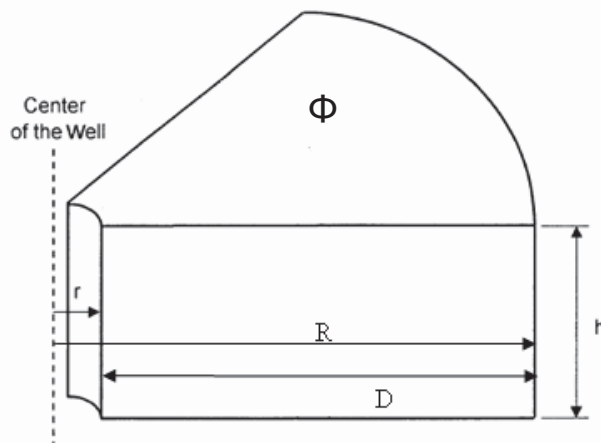


Figure 11: Sketch of resin penetration depth [modified from 10]

- r = wellbore radius
- R = penetration radius (consolidation radius)
- D = penetration depth ($D = R - r$), resin consolidated annulus around the wellbore
- Φ = formation porosity

In an actual wellbore the fluid production is based on radial flow conditions. Within this work the testing to evaluate the suitability of several fluid loss control additives as diverting agents based on the given concept is performed under linear flow conditions. It is a lot easier and cheaper to start feasibility studies on newly developed products under linear flow conditions. This can be regarded as common practice in the industry.

In order to get values for the required resin leak off per unit area of the wellbore a spreadsheet was set up. By defining the wellbore radius and the formation porosity it calculates the required fluid leak off (which in this concept should be the spurt loss) in gal/ft^2 for a range of penetration radii (R).

Figure 12 shows a plot of formation penetration depth (D in ft) versus spurt loss (in gal/ft²) for various wellbore diameters ranging from 5 to 8 inch. The plot is based on a formation porosity of 30% which, as already stated in chapter 1.3, indicates high probability of the need for sand control.

Appendix A includes plots based on formation porosities of 25% and 20%. By comparing the diagrams it can be seen that the required spurt loss for a specific formation penetration depth increases with increasing formation porosity. Further the spurt loss per unit area decreases with increasing wellbore diameter. In below tables the spurt values for 5 and 6 inch wellbores in 30% porous formation are given. Additional tables for wellbore diameters of 7 and 8 inch can be found in Appendix A.

When taking into account all unfavourable factors of a real wellbore situation the requirement of consolidation radius increases from a few inches to about 2-3 feet. ^[1] To achieve a consolidated radius of 2ft in a 5in open hole completion a spurt loss of 21 gal/ft² would be needed (formation porosity 30%; Table 4). The used HPHT fluid loss cell has a working volume of 493ml and considering that fluid has to remain in the cell to prove actual fluid loss ability of the specific product concentration a spurt loss of about 470ml (resulting in about 4.59gal/ft²) is considered to be the highest value achievable within this testing device. This is valid for testing on Fann filter discs of various pore sizes. For consolidation tests a Teflon sleeve sand consolidation chamber has been added to the HPHT cell. The inlet valve of the consolidation chamber is attached to the HPHT cell discharge valve. In this case the filtering area is reduced and higher spurt values (in gal/ft²) can be reached with the same working volume. Reduction of filtering area means that the surface area of the sand pack is smaller than the filtering area of the sand disc. The actual sand consolidation work will be discussed in chapter 4.

This testing series is the first step to get an idea about the products behaviour and it will clarify if more complicated tests are worth conducting with one of the fluid loss additives. Therefore some simplifications had to be made and some actual downhole realities neglected. These assumptions are summarized in the table below.

Reality	Simplification
Radial flow in the wellbore	Linear flow conditions in HPHT cell
Connate water saturation	For calculations of penetration depth the water saturation has been neglected
Cased hole completion	For the calculation of penetration depth openhole completion was assumed

Table 3: Simplifies testing conditions

Within this chapter it will be evaluated if the tested fluid loss products are suitable to achieve a high spurt loss and therefore allow resin transportation into the formation to result in adequate consolidated strength to resist drawdown pressure and drag forces from fluid production. Further the influence of permeability on the achieved spurt loss will be determined by testing on various filter discs.

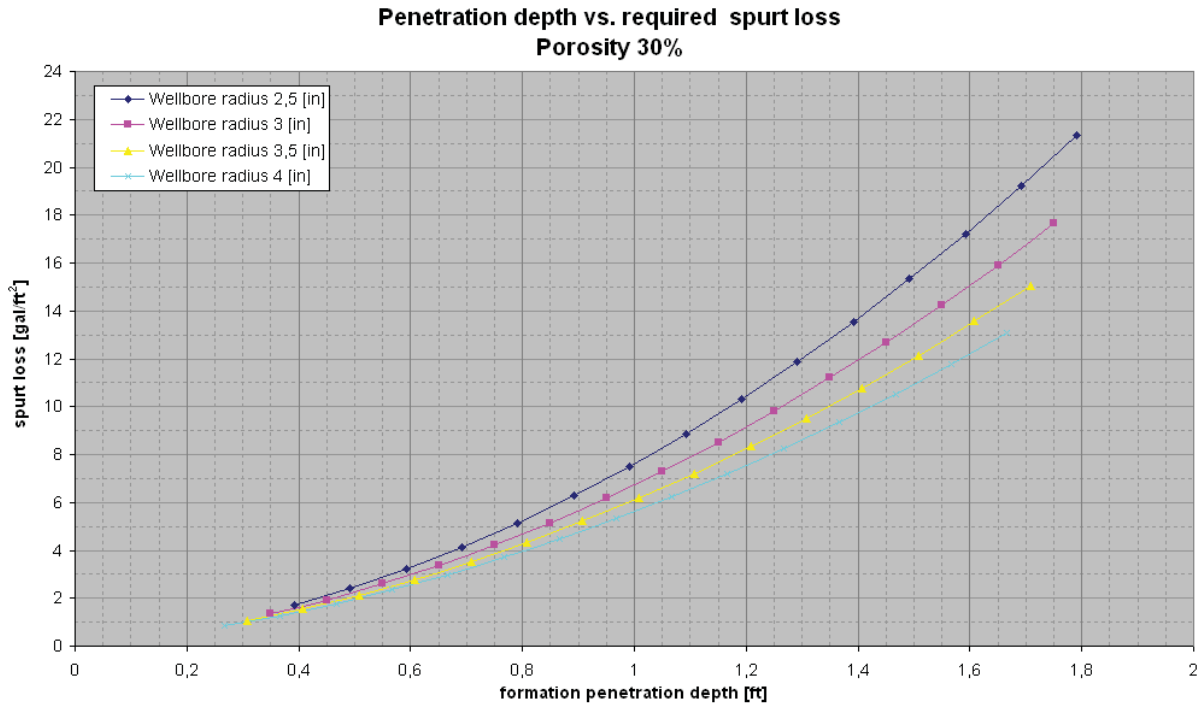


Figure 12: Penetration depth vs. required spurt loss (formation porosity 30%)

Penetration radius [ft]	penetration depth [ft]	spurt [gal/ft²]
0.6	0.39166667	1.705066
0.7	0.49166667	2.405194
0.8	0.59166667	3.213034
0.9	0.69166667	4.128586
1	0.79166667	5.15185
1.1	0.89166667	6.282826
1.2	0.99166667	7.521514
1.3	1.09166667	8.867914
1.4	1.19166667	10.322026
1.5	1.29166667	11.88385
1.6	1.39166667	13.553386
1.7	1.49166667	15.330634
1.8	1.59166667	17.215594
1.9	1.69166667	19.208266
2	1.79166667	21.30865

Table 4: Required spurt loss (wellbore diameter 5 [in], $\Phi=30\%$)

Penetration radius [ft]	penetration depth [ft]	spurt [gal/ft²]
0.6	0.35	1.33518
0.7	0.45	1.91862
0.8	0.55	2.59182
0.9	0.65	3.35478
1	0.75	4.2075
1.1	0.85	5.14998
1.2	0.95	6.18222
1.3	1.05	7.30422
1.4	1.15	8.51598
1.5	1.25	9.8175
1.6	1.35	11.20878
1.7	1.45	12.68982
1.8	1.55	14.26062
1.9	1.65	15.92118
2	1.75	17.6715

Table 5: Required spurt loss (wellbore diameter 6 [in], $\Phi=30\%$)

3.1.1 Evaluation of test data

During an HPHT fluid loss test the fluid loss over time is recorded and the obtained data is evaluated as follows. The cumulative filtrate volume is plotted versus the square root of time (in minutes). In the figure below it can be seen that after an instantaneous fluid loss occurring after opening of the discharge valve (spurt loss) a filter cake is built up and the fluid loss almost stops. During this particular test the fluid loss remained constantly low over 63 minutes. In case the fluid breaks through the developed filter cake after a certain period of time the plot would show an exponential increase of cumulative fluid output over square root of time.

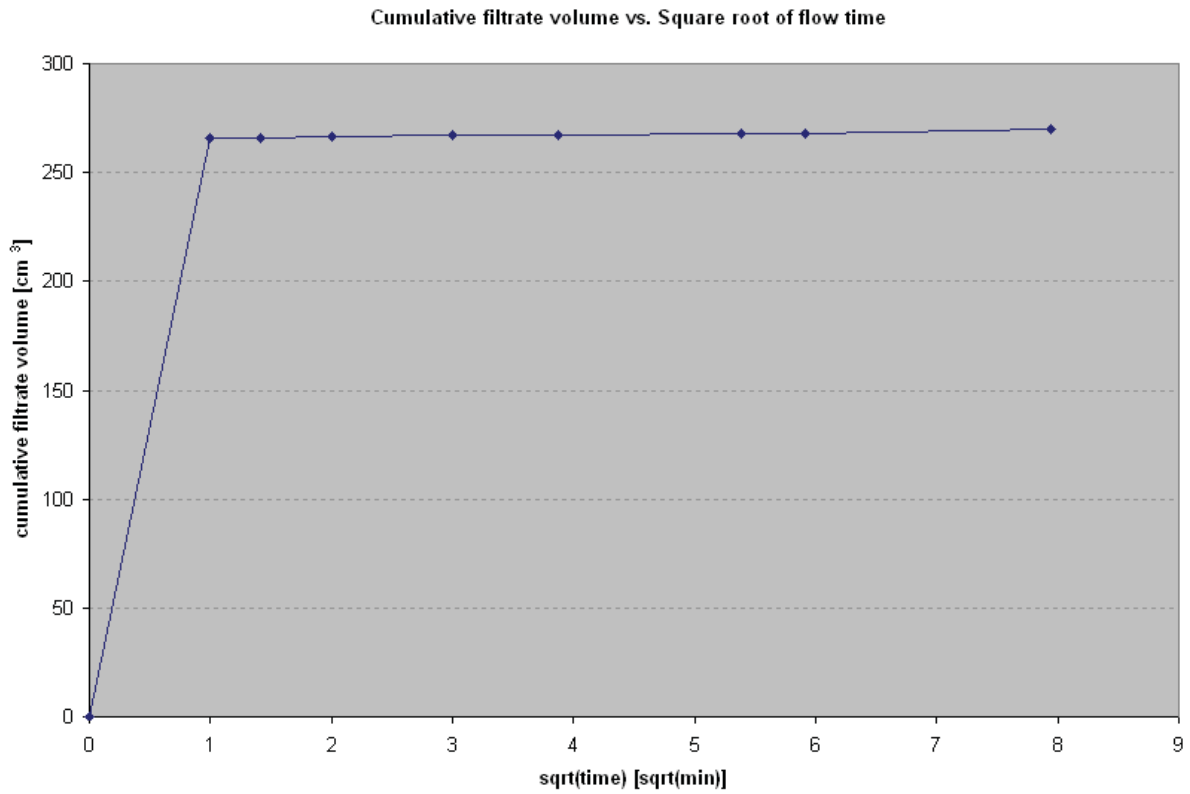


Figure 13: Cumulative filtrate volume vs. sqrt(time)

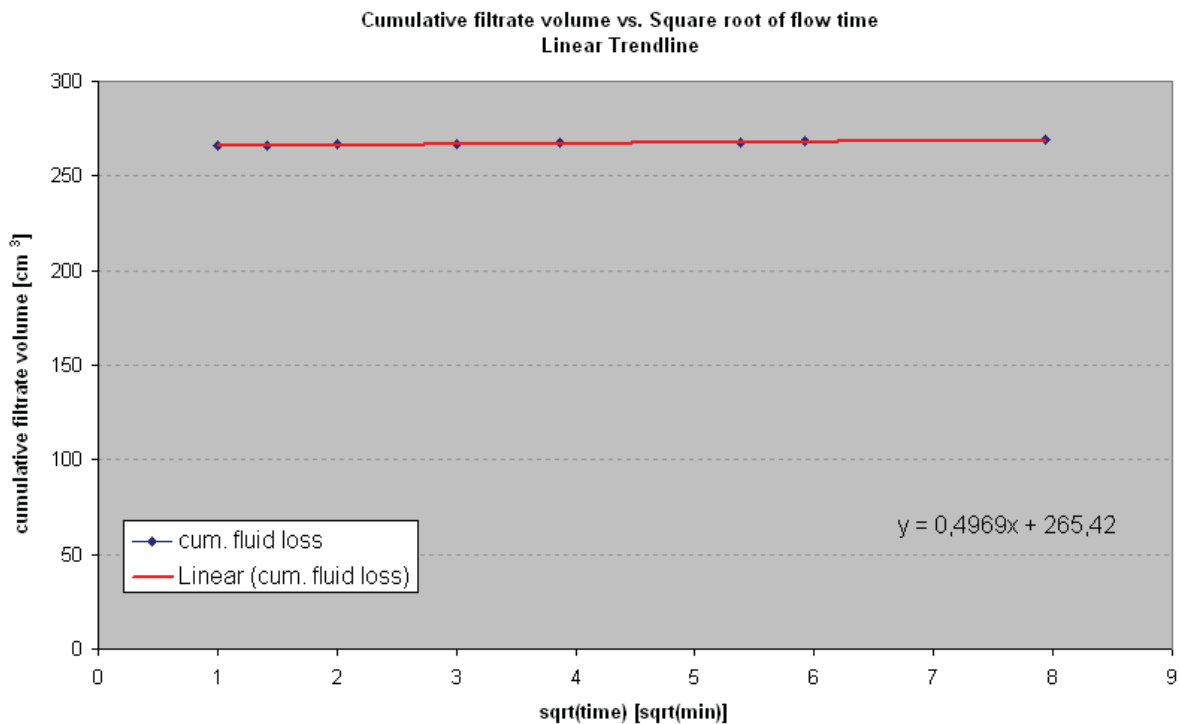


Figure 14: Linear trendline of cumulative filtrate volume vs. sqrt(time)

Afterwards a plot showing only the data points recorded after the filter cake had been built up (Figure 14). A linear trendline is added to the data points and its formula is displayed on the plot. This linear equation ($y = k \cdot x + d$) gives the slope (k respectively m) of the straight line and its intersection (d) with the ordinate. In the given example the slope of the linear trendline is 0.4969

$\text{cm}^3/\text{min}^{1/2}$. The intersection of trendline and ordinate gives the spurt loss in cm^3 . With these values determined by laboratory testing further calculations can be made.

The fluid loss characteristics of a wall-building fluid can be described by using its fluid loss coefficient. To calculate the wall-building fluid loss coefficient the filtration area and the slope of the fluid loss curve are needed.

$$K_w = 0.0328 \frac{m}{2A} \quad (\text{Eq.3.1})^{[11, 12]}$$

K_w or C_w = wall building fluid loss coefficient [$\text{ft}/\text{min}^{1/2}$]

m = slope of fluid loss curve, plotting cumulative filtrate volume from a filter press vs. the square root of flow time [$\text{ml}/\text{min}^{1/2}$], [$\text{cm}^3/\text{min}^{1/2}$]

A = cross-sectional area of test media through which flow takes place [cm^2]

$$K_w = \frac{m}{2A} = \frac{\frac{\text{cm}^3}{\sqrt{\text{min}}}}{2\text{cm}^2} = \frac{\text{cm}}{2\sqrt{\text{min}}} = \frac{0.0328 \text{ ft}}{2\sqrt{\text{min}}} = 0.0164 \frac{\sqrt{\text{min}}}{\text{cm}^2} = \frac{\text{ft}}{\sqrt{\text{min}}}$$

Using a filtration area of 25.108cm^2 equation 1 gives a fluid loss coefficient of $0.0003246\text{ft}/\text{min}^{1/2}$ for this particular example. A low K_w value stands for small fluid leak off and good sealing ability of the fluid loss additive. It indicates good performance of the wall-building fluid, which means the creation of a sealing filter cake. In general values for K_w being 0.006 or above are considered to be high; a K_w value of 0.005 would be acceptable. Of course the lower a K_w value the better the fluid loss control of the particular product; a value of 0.000xy shows very good fluid loss control over time.

In order to get an idea of the spurt loss per unit area the spurt loss volume is divided by the filtering area. For the above example this calculation gives a spurt loss per unit area of $2.594\text{gal}/\text{ft}^2$.

$$\frac{\text{spurt loss}}{\text{unit area}} = \frac{\text{cm}^3}{\text{cm}^2} = \text{cm} * 0.0328 = \text{ft} = 7.48 * \frac{\text{ft}^3}{\text{ft}^2} = \frac{\text{gal}}{\text{ft}^2}$$

In order to be able to compare the results gained from the fluid loss tests also the testing environment (pressure and temperature), concentration of fluid loss product, KCl concentration, alteration of pH value and the pore size of the ceramic filter discs used are recorded.

3.2 Testing Equipment and procedures

This section gives an overview of the HPHT equipment used for fluid loss testing and describes the procedure of an HPHT test. Further the set up and conduction of screening test is outlined.

3.2.1 HPHT Equipment

Pressure and temperature encountered in a wellbore can influence the behaviour of a fluid loss additive and its interaction with the carrier fluid and the formation sand. In order to simulate high pressures and temperatures the fluid loss tests were conducted in an HPHT (High-Pressure, High-Temperature) filter press series 387. The filter press used was manufactured by the Fann Instrument Company, Houston, Texas.

The maximum working pressure of this cell is 1800psi and testing up to a temperature of 500°F is possible. The specifications of the Fann Filter Press are given in Table 6.

Maximum Working Pressure	1800 psi
Maximum Temperature	500°F
Power Requirement	115/230VAC 50/60 HZ
Sample Cell Volume	493 ml
Heating Capacity	800 watts
Filtering Area	22.6 cm ² (3.5 in ²)

Table 6: Specifications of Fann Filter Press [14, 15]

The double ended 500ml cell was adapted for the use of Fann ceramic filter discs (thickness 1/4"), therefore the actual cell volume is reduced to 493ml.

Instead of using the original Fann cell cap at the upper end of the cell body a cap with an attached adapter for a stirring device was built by the Halliburton technicians. This stirring device allows keeping the fluid loss particles in suspension and prevents premature settling of the particles on the filter disc surface.



Figure 15: HPHT Filter Press



Figure 16: Parts of the HPHT Filter Press

Ceramic filter discs manufactured by Fann Instrument Company were used as filtering material. The discs are available in several mean pore diameter sizes (5, 10, 20 and 35 micron) and have a diameter of 2 ½” and their thickness is ¼”.

Measurements showed that the filtering area given at the specifications of the Fann filter press is slightly smaller than the measured area of the actual filter cake. For the data evaluation the area of the actual filter cake has been used. This difference might appear because the O-ring on the bottom provides the actual seal on the ceramic filter disc. The filter area measured by the manufacturer is calculated using the inner diameter of the cell body which is about 0.1inch smaller than the actual filter cake diameter. Therefore the resulting filtering area used for calculations has been 3.892in² instead of 3.5in².

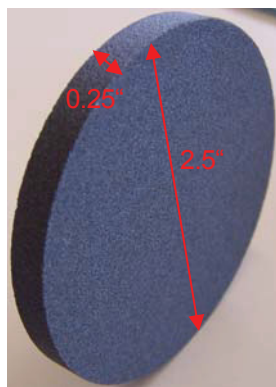


Figure 17: Fann filter disc – mean pore diameter 20 microns



Figure 18: HPHT cell body- top view

For most tests the 20 micron ceramic filter disc was used. When saturated with water the 20 micron discs weigh 50.85g compared to the dry discs which weigh 44.03g.

3.2.2 HPHT test procedure

To conduct a fluid loss test the desired pressure and temperature has to be adjusted on the HPHT device. Depending on the temperature chosen it may take some time to heat up the cell. A ceramic filter disc of desired pore diameter is inserted in the lower end of the body cell and the bottom cell cap is screwed on the body cell. Afterwards the body cell is put into the HPHT filter press. Subsequently a carrier fluid system (brine or resin) is poured into the body cell. The fluid loss product is either added after pouring the fluid system in the testing device. In case of testing FDP-S875 the cell temperature and the time given the product to soak will affect the results of the fluid loss test. After the mixture of fluid system and fluid loss additive is in the body cell it is agitated manually to keep the particles from settling before the top cell cap with the stirring device adapter is fixed up. The stirring device keeps the fluid loss product in suspension. Its desired RPM is set by using the stirring device control panel (Figure 15). After switching on the stirring device the cap is screwed onto the body cell and the hose providing the pressure supply is connected to the adapter. The body cell is now totally lowered into the heating chamber and pressure is applied. After the cell is fully pressurized the discharge valve on the bottom cap is opened and fluid outflow over time is recorded by using a stopwatch. After the spurt loss is finished and the fluid loss additive starts to seal off the disc area the stirring device is switched off. Fluid output is recorded from 16 minutes up to an hour.

Generally spoken once a certain product concentration is able to establish a seal on the sand disc it should be able to maintain it. During some tests the fluid broke through the developed filter cake. But this is not considered to be a concern in an actual diversion job. In a real downhole situation fluid containing the fluid loss additive is pumped past the formation continuously. Therefore a leak would be repaired by deposition of more fluid loss product.

After finishing recording of fluid output over time the discharge valve is closed and the pressure is turned off. After turning off the pressure supply the pressure in the body cell is blown off and the pressure hose can be disconnected from the adapter. The body cell is elevated with a lifting device and the top cap is screwed off the body cell. Finally the filter disc is removed by unscrew the bottom cap. Before running the next test the body cell is cleaned.

3.2.3 Method used to evaluate basic product behaviour

In order to get an idea of the products behaviour and filter cake texture at different temperatures the following test procedure has been created. A disposable filter funnel by Whatman is mounted on a Pyrex filter flask. Filter papers of different micron size can be put into the filter funnel in order to create various test beds. To keep the fluid loss particles in suspension a stirring device is added to the testing equipment. The filter flask is connected to the house vacuum line of the Halliburton Technology Center. The suction pressure provided by the vacuum line may slightly vary between 14 and 16 psi. The described set up provides a good screening possibility because product behaviour can be evaluated without using the expensive ceramic filter discs. Further the filter cake textures of different product phases (wet product, dry product) and grain sizes at various temperatures can be analysed. Apart from that the compatibility of fluid loss additive and resin system is tested this way, because it would be a waste of products (ceramic discs and resin) to conduct those basic screening test in the HPHT cell. Variation of the filter papers (25 micron, 11 micron, 8 micron) provides several permeabilities for the testing.

To conduct the test a mixture of carrier fluid and fluid loss additive is poured into the funnel while the stirring device is already switched on. Then the vacuum pressure is applied and the fluid output is recorded. For this test procedure recording the fluid output means measuring the time needed for 200ml of brine to flow through the filter paper into the filter flask. The fluid system is stirred during the entire test.



Figure 19: Device to evaluate basic product behaviour

3.2.4 Testing of Diverting Agents

Previously in this chapter the diversion concept, the data evaluation process and the equipment have been described. The objective of ongoing testing which will continue this work is to test the diversion concept and the suitable products in a Hassler device to define its diversion effectiveness on various formation permeabilities. Before this is going to be done more basic tests on product behaviour, fluid loss control abilities and achievable spurt values of all three products in question have to be tested. In case one of the products shows its suitability regarding the mentioned prerequisites as well as shows good and similar performance over a permeability range tests may continue in a Hassler device.

Further the title of this thesis defines that methods for diversion of resins should be found. Within chapter three tests performed with KCl brine as fluid system are described. It has been used as carrier fluid to figure out best suitable temperature and fluid loss control additive concentration before switching to the more expensive resin system. Further handling of KCl brine is easier and not as time consuming as testing resin in an HPHT cell. The composition of the resin involves major clean up of the discharge valve, cell body and bottom cap of the HPHT cell. If one or more products would prove to be suitable for this concept further testing regarding compatibility of the product with a water based resin system would be tested and describes within chapter 4.

The following chapters describe the testing of three different fluid loss additives for its suitability for the defined concept. A filter cake and its sealing ability as well as the spurt loss are influenced by various factors as for example the concentration of a fluid loss additive, the pressure and temperature conditions as well as the porosity and permeability of the filter medium and the composition of the carrier fluid.

3.3 Testing of FDP-S875-07 as a Diverting Agent

The first fluid loss additive tested as diversion agent based on the defined concept is FDP-S875-07. This product is a future development product of Halliburton and has not been tested very much. It is available in wet and dehydrated form; especially the dehydrated product version has not been tested much. The product has been developed in 2007 and is still at the beginning of testing and is

not yet sold by Halliburton. The used dehydrated version of FDP-S875 is manufactured by the Halliburton supplier Champion.

The fluid loss additive FDP-S875 is part of the chemical family of inorganic salt polysaccharide solutions. It is composed of hydroxypropyl guar (HPG) and potassium chloride. As mentioned in chapter 2.3 hydroxypropyl guar is a derivative of the guar gum.

Derivatized guar is less suitable as nutrition for humans but it is less sensitive to biological degradation. Hydroxypropylation in particular increases the solubility of the gum which results in rapid hydration regardless of water temperature. HPG has improved electrolyte capability, less hydrogen bonding tendency as well as increased solubility in alcohol/water solutions. ^[22] In FDP-S875 the HPG is cross-linked with borate in order to form guar polymer chains resulting in a sealing gel.

Diversion should take place temporarily; therefore one of the most important issues is the removal of a diversion agent without damaging the formation. The filter cakes created by S875 can be degraded by reducing its pH, which means an acid treatment would be suitable to re-establish permeability.



Figure 20: Guar beans

FDP-S875 is available as wet product as well as dehydrated and can be grind to smaller grain sizes. Testing started out with the wet product and was successful only by using 10g of product. As it seemed to be hard to create a seal with less amount of S875 and the lab offered the possibility to grind the dehydrated product the testing was focused on the dehydrated FDP-S875 product.

3.3.1 Basic product behaviour

This chapter gives an overview of basic product behaviour of FDP-S875. As especially the dehydrated S875 has not been tested often it was necessary to record behaviour as hydration at various temperatures, particle size used for testing as well as sealing abilities on filter papers under varying temperature and test bed conditions.

3.3.1.1 Hydration Tests

Hydration tests of coarse dehydrated and wet FDP-S875 were performed to determine the time frame available before particle swelling occurs. Five samples of FDP-S875 in 100ml 6% KCl brine were prepared.

Sample number one contains 3g of dehydrated coarse product in 100ml 6% KCl brine at room temperature. After 30 minutes no visible particle expansion occurred. The 3 grams of fluid loss additive filled 15ml of the jar. After 30 hours the product occupied 50ml of the jar and formed a jelly-like mass. No free particles remained in the water. The hydration of the particles led to a 3.3-fold increase in particle volume.

Sample number two contains 6g of wet product in 100ml 6% KCl brine at room temperature. After half an hour no visible particle swelling has been observed. As in sample number one the 6 grams

of wet product filled about 15ml of the jar. After 30 hours the product occupied 40ml of the jar and formed a jelly-like mass and just a few free particles remained in the water. In this case the volume increased by a factor 2.6.

Sample number three contains 3g of dry product in 100 milliliters 6% KCl brine at 150°F. After inserting the fluid loss additive the sample was able to cool down and reach room temperature. At 150°F the particle swelling and clumping started right away. After shaking and stirring the particles could be separated and settled down again. Five minutes later they clumped together once more and could be lifted as a little ball. After about half an hour the product occupied already 55ml of the jar. The sample stayed at room temperature over night and thirty hours later 18ml of remaining free water could be poured out of the jar. The jelly-like mass now occupied 80ml of the jar volume. Taking into account that 3g of FDP-S875 fill 15ml of the jar it can be stated that the particle volume increased by a factor 5.3. Sample three showed the highest volume increase out of these five samples. Beside the fact that this sample cooled down to RT this particular jar has been shaken more often than the other ones.

Sample number four contains 3g of dry product in 100ml 6% KCl brine at 150°F. The sample has been put into a 150°F water bath over night. After 28 hours the jelly-like mass occupied only 35ml of the jar compared to the 50ml of sample number one which remained at room temperature. Apart from the gel on the bottom of the jar free clumps were left in the brine.

Sample number five contains 6g of wet product in 100ml 6% KCl brine at 150°F. The sample has been put into a 150°F water bath over night bath. After 28 hours the jelly-like mass occupied only 15ml of the jar compared to the 40ml of sample 2. Apart from the gel on the bottom of the jar also free clumps were left in the brine.

The following data is collected by hydrating the FDP-S875 particles ground by using a 0.25 mm screen in the Cyclone mill (for more details see chapter 3.4.1.2). For this hydration test FDP-S875 has been put in jars filled with 100ml 6% KCl brine at different temperatures. The results are summarized in the table below.

Particle size d (0.5) = 105.048 micron	10 minutes	23 minutes	27 minutes	36 minutes
Room temperature (70°F)	no signs of swelling	no signs of swelling or clumping together	smaller particles show signs of swelling	By putting particles in motion (no hard stirring – just lifting grains from bottom of the jar) it could be seen that particle accumulations had formed. The clumps can be destroyed by stirring. After settling of the particles and bringing them in motion once again clumps will have formed again.
115°F	After 3 minutes the particles small enough to stay in suspension have already swollen. Particles start to form small accumulations and clump together. The clumps can be destroyed by stirring. After settling of the particles and bringing them in motion once again clumps will have formed again.			
193°F	The particles swell immediately and start forming clumps.			

Table 7: Hydration behaviour of dehydrated particles (0.25 mm grind) in 6% KCl

Table 7 shows that at room temperature it takes longer for the particles to swell and clump together. Increasing the temperature by 45°F (from RT to 115°F) reduces the time before swelling of the grains starts from about 27 minutes to 3 minutes, which means a 9-fold reduction in time before the particles show signs of swelling could be observed.

3.3.1.2 Particle Size

George Suman Jr. et al. give a guideline for a close filter cake packing in order to reduce its permeability and porosity. The median bridging-particle size (at least 5 Vol %) should be equal or slightly larger than one-third median pore size of the rock. The particle size distribution should be wide and particles approaching colloid size should be included. [6]

Keeping in mind that at room temperature particle swelling starts after about 30 minutes it was intended to create various particle size distributions by grinding the dehydrated coarser product delivered by Champion with a Cyclone mill (Figure 21). So apart from cross linking and forming a seal at elevated temperature the intention was also to create a filter cake consisting of a close pack of S875 particles at room temperature.

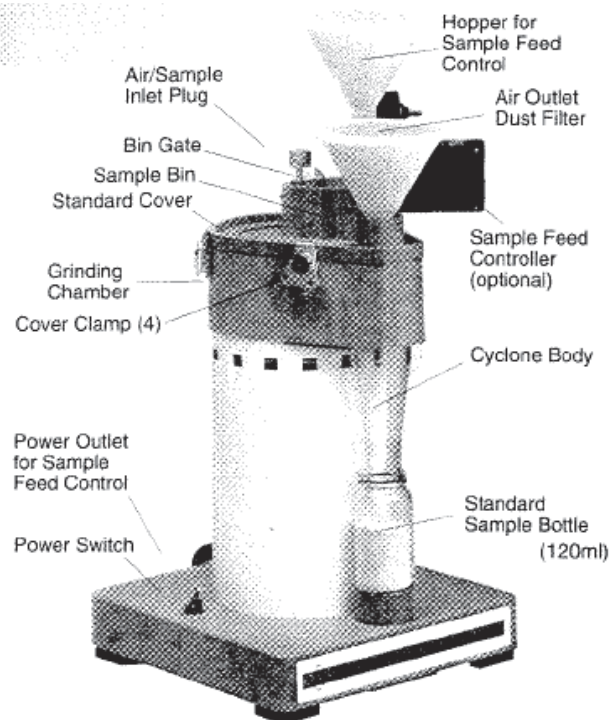


Figure 21: Cyclone Sample Mill

A coarser dehydrated grind produced by Champion has been fed into the Cyclone mill. Various screen sizes were used because it was expected that there would be major difference between the 0.25 mm grind and the 2 mm grind. In order to get a wide particle size distribution the intention was to mix the four grinds. As Table 8 shows the average particle size $d(0.5)$ of the various grinds does not vary as much as expected. Over those the median particle size is larger way larger than one-third of the median pore diameter of the sand discs used for testing.

	$d(0.1)$ [micron]	$d(0.5)$ [micron]	$d(0.9)$ [micron]
Screen size 0.25 mm	10.855	105.048	301.893
Screen size 0.4 mm	16.946	143.511	356.508
Screen size 0.8 mm	17.509	153.592	369.144
Screen size 2 mm	21.221	168.455	381.434

Table 8: Particle size distribution of various cyclone mill grinds

As above particle size distribution shows it was not possible to create a median particle size as small as about one-third (1.6, 3.3, 6.6, and 11.6 micron) of the various filter discs available (5, 10, 20 and 35 micron) by using the cyclone mill. This explains why the main part of the subsequent testing has been performed at elevated temperatures in order to use the products ability to cross link and form a gel instead of creating a dense pack defined by its particle size distribution.

The particle size distribution has been determined by the analytical department of the Technology Center. The particles produced with the 0.25 mm screen are the smallest and are those used the most in the subsequent HPHT tests. The particle size distribution of this grind is shown in Figure 22, plots showing the distribution of the other screen openings (0.4, 0.8 and 2 mm) can be found in Appendix B.

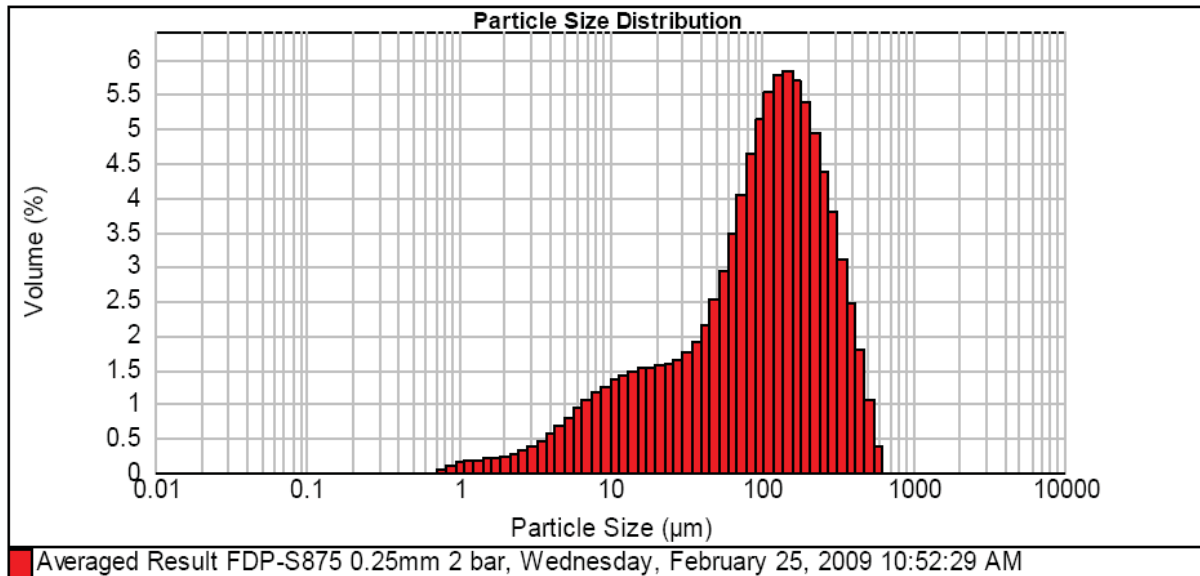


Figure 22: Particle size distribution of FDP-S875 cyclone mill grind (screen size 0.25 mm)

According to the Cyclone mill data sheet one of the advantages of sample creation with this mill is its consistency in particle size creation. Exactly this point is apart from the fact that a 0.25 mm screen creates too large particles anyway a downside for this specific purpose. Therefore considering the wide size distribution and small grains necessary for a packed and non cross linked filter cake the cyclone mill is not the ideal machine to grind the product for this purpose.

3.3.1.3 Testing of basic product behaviour on filter paper

Before grinding FDP-S875 the dehydrated coarser particles as well as the wet particles had been tested on Whatman filter papers using the device described in chapter 3.2.3 without the stirring device. The FDP-S875 filter cakes on the subsequent images are created on 8 micron filter papers after putting the product in to 6% KCL brine for 5 minutes (soaking time of the S875). The images are supposed to show the difference in filter cake textures depending on temperature.



Figure 23: dry FDP-S875 filter cake on 8 micron filter paper (180°F)



Figure 24: dry FDP-S875 filter cake on 8 micron filter paper (RT)

The first filter cake (Figure 23), created at 180°F with the dehydrated S875 is a jelly-like, elastic filter cake. Cross linking of the particles allows the formation of a gel. No single particles can be seen by looking at the cakes. The same is true for the filter cake made from the wet FDP-S875 version (Figure 25). The filter cakes created at high temperature showed expansion immediately after taking them out of the filter device. Further expansion could be seen the next day. The sealing ability of these cakes is extremely good, after a spurt loss the gel sealed off the filter paper.

The filter cakes created at room temperature (Figure 24 and 26) are brittle and product particles are still visible within the cake. As it can be seen by comparing the mentioned images the difference between wet and dry product is the particle size. The cake created from the dry product is thinner and seems to be denser due to the smaller and therefore less hydrated particles. None of these cakes showed any expansion after removing it from the disposable funnel used for testing. Fluid flow through the filter papers could not be stopped by these cakes. After the spurt loss the fluid throughput was reduced but no shut off was reached. Brine passed through the pore space of the grain pack until the 200ml of 6% KCl brine were removed from the funnel.



Figure 25: wet FDP-S875 filter cake on 8 micron filter paper (160°F)



Figure 26: wet FDP-S875 filter cake on 8 micron filter paper (RT)

Concerning the sealing ability the same is true for cakes created at the 25 and 11 micron filter papers. Further the filter cakes showed the same textures when testing FDP-S875 on other test beds.

How these various filter cake textures affect the flow rate through the filter papers is displayed in the diagram below. The wet and the dry fluid loss additives were tested on all available filter papers at room temperature as well as at elevated temperature (150°F). The images of the filter cakes created at room temperature already indicate worse sealing ability compared to the cakes made at higher temperature. This can be confirmed by Figure 27.

For comparison it should be mentioned that the flow rate through a 25 micron filter paper is 100 ml/sec and for the 11 micron filter paper it is 50 ml/sec. So even though not being able to grind the optimal particle size distribution for creating a dense filter cake at room temperature it was possible to reduce the flow rate to about 0.3 ml/sec for all filter papers. At 150°F the flow rate through the filter papers could be reduced to about 0.05 ml/sec. There is not much difference by comparing the performance of dry and wet S875. At 150°F the wet product reduced the flow rate slightly more than the dry one. At room temperature on the other hand the dry product created a better filter cake leading to a lower flow rate. This slightly better performance of the dry product can be explained by the difference in filter cake texture which shows the denser pack of smaller particles formed by the dry S875 at room temperature. In general it can be stated that the sealing performance of FDP-S875 is better at elevated temperatures. As mentioned at the beginning of this chapter no stirring device was used for creating these filter cakes and by collecting the data displayed in Figure 27. For the subsequent testing recorded in Figure 28 and 31 a stirring device has been added to the testing device. This explains the order of magnitude difference in flow rate between Figure 26 and 28. Due to the stirring the FDP-S875 particles are kept in suspension and the filter cake build up is delayed leading to faster fluid loss. Further the test shown in Figure 27 was performed by using the

dehydrated coarser product as delivered by Champion. For the following testing the cyclone mill grinds are used. These particles are smaller and therefore easier to keep suspended.

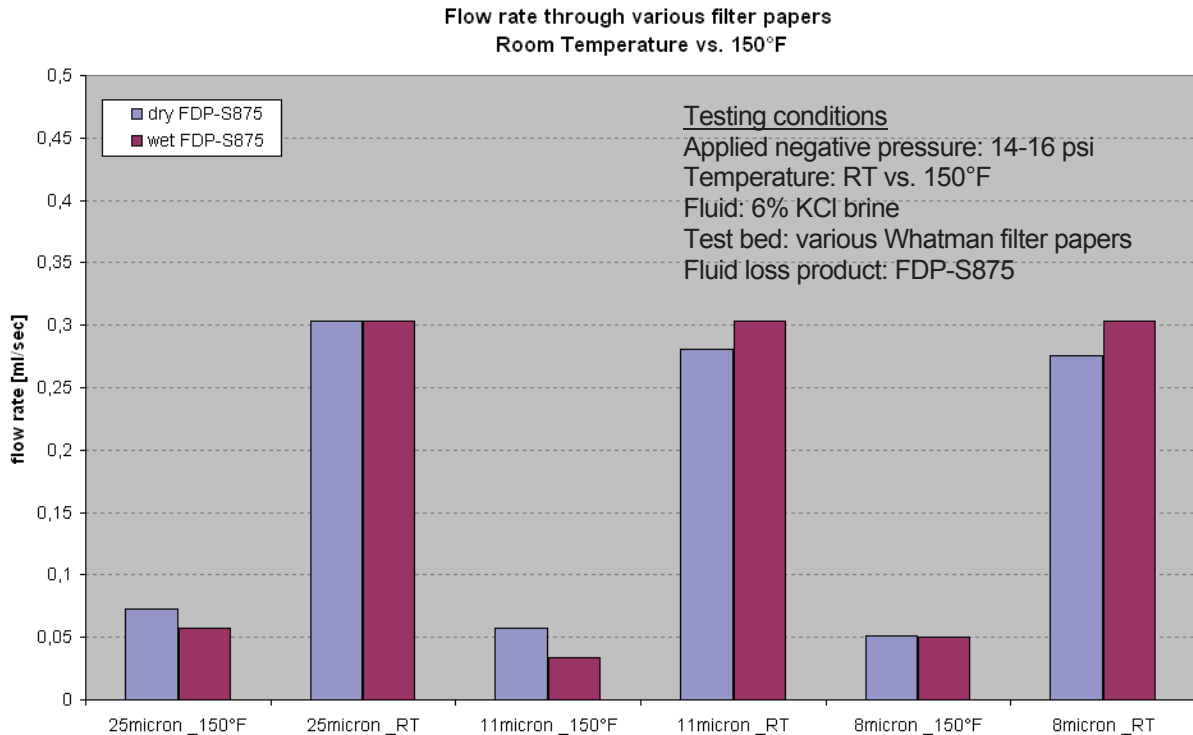


Figure 27: Flow rate through various filter papers comparison RT vs. 150°F

Figure 28 shows the results of testing the various grinds given in Table 8 on different filter papers at room temperature. The product concentration used was held constant at 0.015 g/ml. The testing device and procedure described in chapter 3.2.3 was used for the tests. The flow rates obtained at the 11 and 8 micron filter paper do not vary a lot with change in particle size.

For all grind sizes the flow rate on the 11 respectively 8 micron paper lies between about 1 and 1.5 ml/sec. Only on the 25 micron filter paper test bed the grind sizes show greater variation in flow rate. The 0.8 mm screen grind created the filter cake with the highest flow rate (3.2ml/sec) and the 2mm screen grind resulted in a filter cake giving a flow rate of 2.9ml/sec. While for the other grinds the flow rate on the 25 micron filter paper is significantly higher compared to the 11 and 8 micron paper it is not the case for the smallest grind. The particles ground with the 0.25mm screen show similar results on all three filter papers with a trend of decreasing flow rate with reduction in filter paper pore diameter.

Of course certain randomness in particle settlement has to be taken into account. This settlement and subsequent filter cake build up may lead to more or less void spaces in the filter cake leading to higher or lower porosity and permeability of a filter cake.

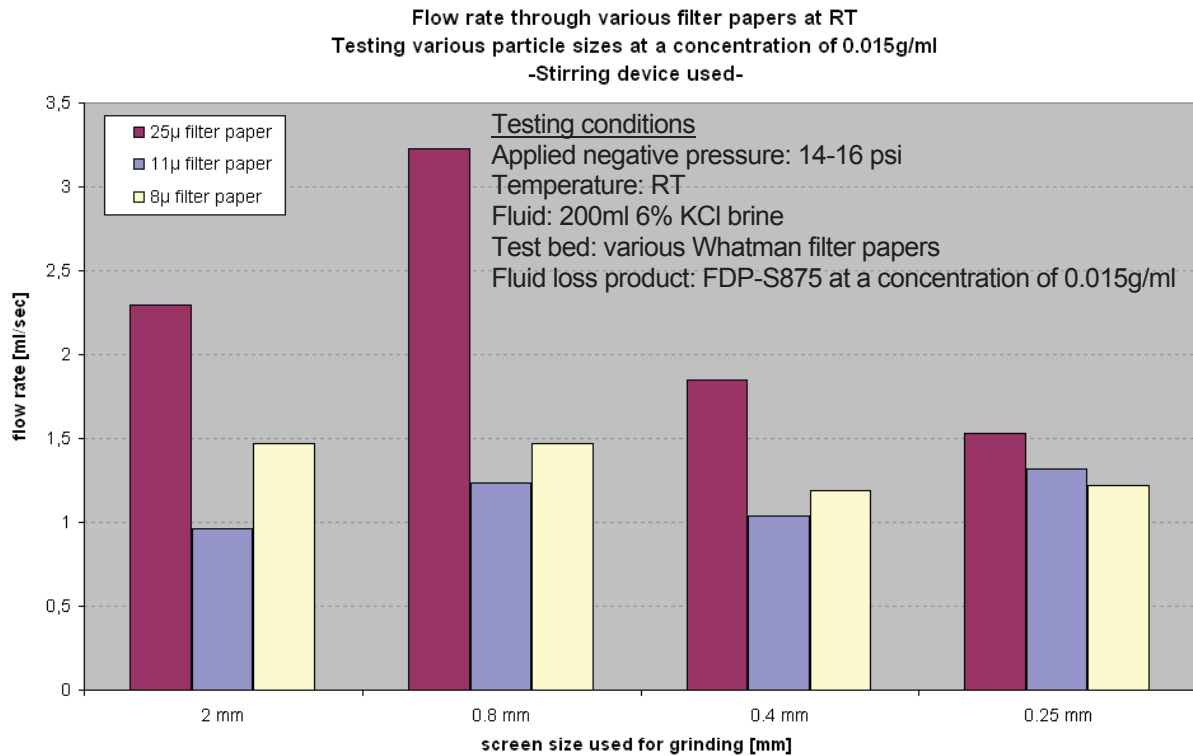


Figure 28: Testing various FDP-S875 grinds as fluid loss additive at RT

During the testing series on the filter papers just brittle filter cakes have been created at room temperature. At RT the 200ml of brine are lost too quickly to give the particles time to cross link. Therefore 200 ml of water were poured on a filter cake created on RT (the texture of the filter cake was as shown in Figure 26: grains visible, brittle) and pressure from house vacuum line has been applied. As the filter cakes created at room temperature do not provide total shut off water started to flow through the filter paper. About three quarter of the added water had been lost when the fluid level stopped decreasing. A total seal has been provided. After 32 minutes the filter cake has been removed from the funnel (Figure 29). The resulting filter cake still showed its original texture on the bottom of the cake directly on the filter paper. The FDP-S875 particle grains were still visible. Touching of the cake revealed its brittleness on the bottom. On the surface on the other hand the product particles had cross linked to gel. No more grains can be recognized and no more void spaces provide flow channels for the fluid.



Figure 29: Filter cake showing jelly-like texture at surface and grainy texture on bottom



Figure 30: RT filter cake

Keeping in mind that most of the water had been lost before establishing the seal on top of the filter cake this procedure would create ideal filter cakes after high initial fluid loss which would favour the concept of high spurt loss and low C_w .

Figure 31 shows the results of testing the temperature dependence of the time needed to establish a sealing filter cake. In other words it has been tested how long it takes for the particles to cross link at various temperatures or how much fluid out of 200ml 6% KCl brine is lost before a seal could be created. The tests were performed on 25 micron filter paper. It can be seen that at 178°F only 42ml brine flow through the filter paper before the fluid loss additive seals off the filter paper. At 128°F on the other hand 152ml (about three quarters of the fluid) have been lost before shut off. With 108ml of brine lost the test performed at 143°F lies in the middle. All three tests were performed with a S875 concentration of 0.015 g/ml.

For comparison another test at 147°F was conducted. For this test the product concentration has been reduced to 0.0075 g/ml. At the lowered concentration 126ml were lost before sealing off the filter medium. The lower concentration would therefore lead to a higher spurt loss compared to the 108ml lost at a comparable temperature of 143°F.

The lower the temperature, the higher is the initial fluid loss before sealing off the filter paper. As room temperature on the other hand will not create a seal and therefore is not suitable for the defined concept there has to be a temperature being the borderline between a high initial fluid loss and good fluid loss control over time and the total loss of the fluid at reduced flow rate.

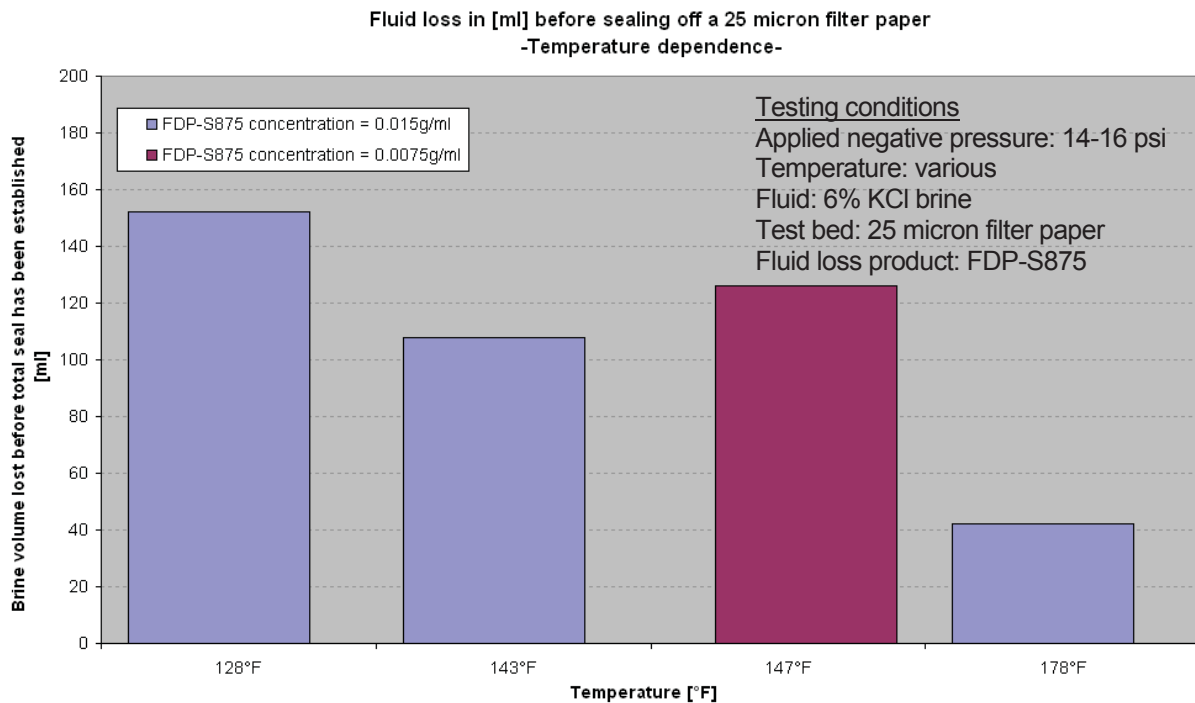


Figure 31: Temperature dependence of fluid flow shut off

It can be concluded that at high temperature the FDP-S875 particles start cross linking immediately. Particles swell and become soft and flexible; they clump together and may settle as little clumps and therefore create a total shut off of fluid flow very quickly. This behaviour leads to good fluid loss control over time (good C_w) but it happens too fast to allow high spurt loss values. At room temperature the established concept cannot be applied because to seal and therefore no resin diversion based on the defined concept can be established. In a real downhole condition room temperature would not be encountered and further bottomhole temperature is most likely to be higher than some of the temperatures subsequent testing will be performed at. Therefore the behaviour of the fluid loss product refers less to the actual wellbore temperature as to figuring out a temperature range in which the diversion concept is able to work from an engineering point of view. This means finding a temperature range at which the desired features of high spurt loss and good fluid loss control can be combined. Once it can be proved that this is possible with S875 chemists would have to figure out a way to apply it over a wide range of downhole temperature scenarios and pumping times.

3.3.2 HPHT fluid loss tests at 200°F

Upfront it has to be mentioned that the HPHT tests at 200°F were performed before evaluating the basic product behaviour described in chapter 3.4.1.3. For these tests the KCl concentration was higher than in the subsequent tests at lower temperature, which means it is easier for the fluid particles to stay in suspension. The HPHT tests were performed according to the description of the test procedure in chapter 3.2.2. This chapter summarizes the most important results obtained from tests at 200°F and 300psi. Figure 32 and 33 show data from fluid loss tests where the coarser dehydrated FDP-S875 was used as delivered by Champion. Figure 34 plots the data recorded from testing the cyclone mill grind (0.25mm screen size).

Figure 32 shows five HPHT fluid loss tests on 20 micron sand discs under before mentioned temperature and pressure conditions. For all tests the concentration of the fluid loss control additive FDP-S875 was 0.015 g/ml. Fluid loss over time was recorded for 16 minutes in four out of the five tests. The last test Feb6_T1 was recorded for 48.5 minutes, it can be seen that after about 34 minutes the fluid breaks through the filter cake leading to the exponential increase of fluid loss over square root of time. By looking at the fluid loss curves it can be recognized that, although the same FDP-S875 concentration was used for all tests, two trends could be established. Test Jan19_T8 (pink graph) and test Jan19_T10 (yellow graph) showed about the same initial fluid loss and therefore resulting in similar numbers for spurt loss (0.64 respectively 0.68 gal/ft²). Also the fluid loss control ability over square root of time represented by C_w is similar for both curves (0.000946 respectively 0.000916 ft/sqrt(time)). The second trend is represented by the three of the five test runs shown as bluish graphs in below diagram. The initial fluid loss lies about 25ml above the other created trend. The calculated spurt loss values range from 0.84 to 0.91gal/ft². The three graphs showing higher spurt loss have slightly worse C_w values (from 0.0018 to 0.0012 ft/sqrt(time)). In case of a fluid breakthrough the data points recorded after the breakthrough are not used for calculations. In test Feb6_T1 the pH value of the brine has been increased to 10. As mentioned before the guar filter cake can be removed by decreasing the pH value, therefore it should be tested if the cake is more stable at higher pH. The light blue graph in below diagram shows that a fluid breakthrough could not be prevented. In general the trend represented by the bluish graphs is favourable for the diversion concept because of the higher spurt loss. The two different trends make clear that even at constant FDP-S875 concentration the spurt loss is not clearly predictable. A certain degree of randomness and velocity of particle deposition has to be taken into account.

So far the tests performed did not vary FDP-S875 concentration. The effect of variation in concentration can be seen in Figure 33. The ideal behaviour would be an increase of spurt loss with decreasing fluid loss control additive concentration. This could lead to a predictable spurt loss and its adjustment by product concentration. In Figure 33 the results of ten fluid loss tests are plotted. The FDP-S875 concentration varies from 0.025g/ml to 0.014g/ml. The first three tests out of this testing series confirm the desired trend. With decreasing the FDP-S875 concentration from test Jan16_T3 to Jan16_T5 the resulting spurt loss increased. The values can be seen in Table 9 as well as Figure 33. Reducing the gel concentration once again by 0.0025g/ml leads to an increase in spurt loss of 0.119 respectively 0.117gal/ft². Further reduction of FDP-S875 concentration to 0.01875g/ml showed a decrease in spurt loss. Ongoing decrease of concentration leads to an increase in spurt loss. Finally despite the difference in concentration the spurt loss values of test Jan16_T5 (0.02g/ml) and Feb6_T1 (0.015g/ml) are similar. Therefore it can be concluded that decreasing FDP-S875 concentration does not necessarily lead to higher spurt loss and better suitability for this concept.

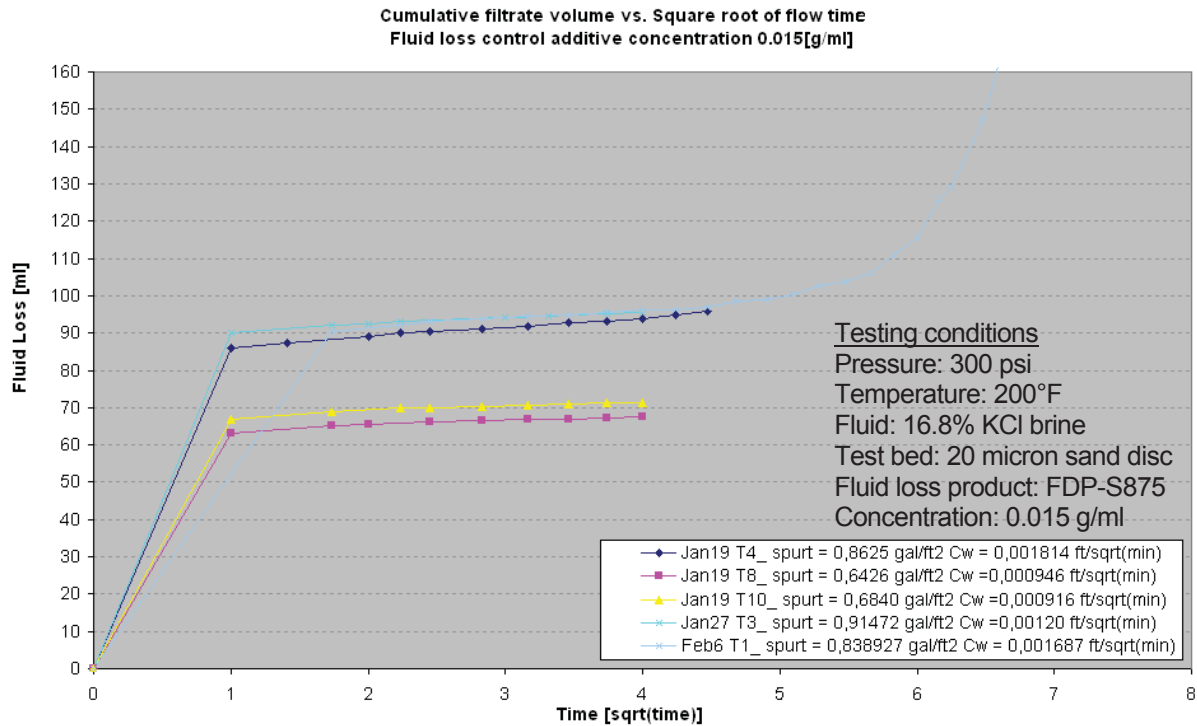


Figure 32: Variation in spurt loss at equal FDP-S875 concentration

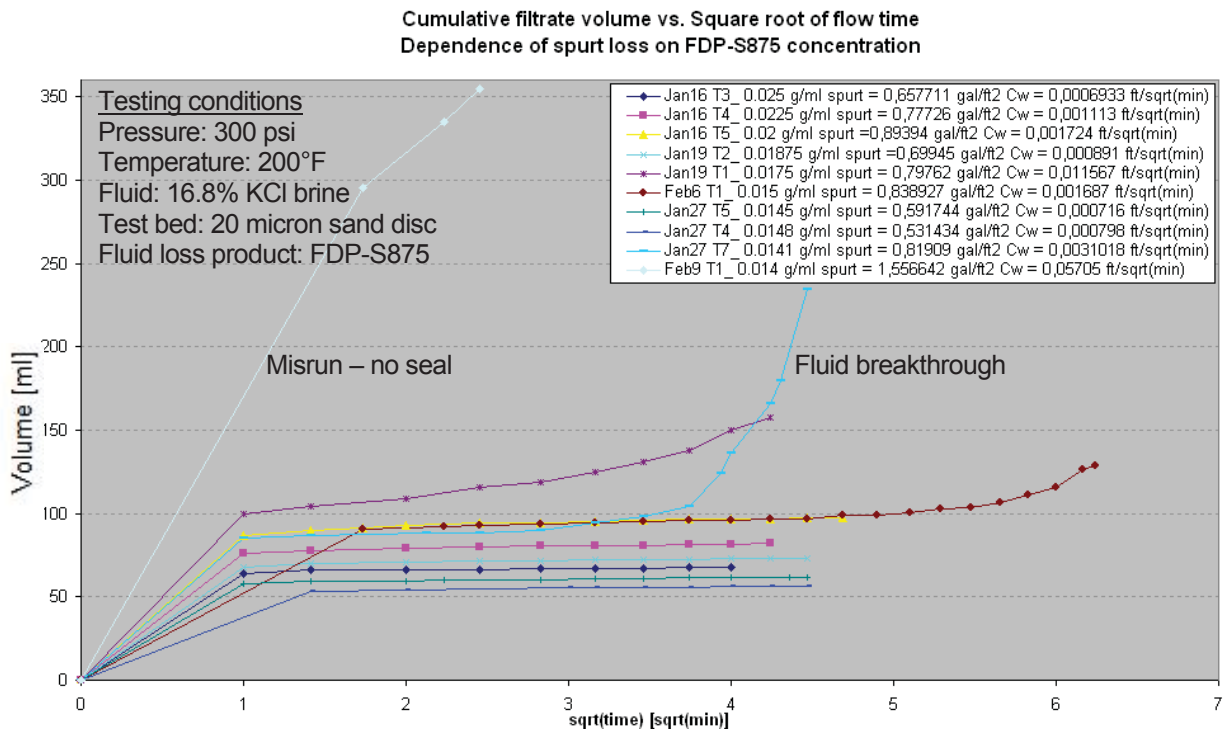


Figure 33: Dependence of spurt loss on FDP-S875 concentration

Test Feb9_T1 had with 0.014g/ml the lowest FDP-S875 concentration. This concentration showed by far the highest initial fluid loss but the fluid throughput did not slow down as much as it would be expected for providing good fluid loss control. But as this concentration is just 0.0001g/ml lower than those of test Jan27_T7 which did create a sealing filter cake the failure of test Feb9_T1 may be an exception.

Test	FDP-S875 concentration [g/ml]	spurt [gal/ft ²]	C _w [ft/sqrt(min)]
Jan16 T3	0.025	0.658	0.0007
Jan16 T4	0.0225	0.777	0.0011
Jan16 T5	0.02	0.894	0.0017
Jan19 T2	0.01875	0.699	0.0009
Jan19 T1	0.0175	0.798	0.0116
Feb6 T1	0.015	0.839	0.0017
Jan27 T5	0.0145	0.592	0.0007
Jan27 T4	0.0148	0.531	0.0008
Jan27 T7	0.0141	0.819	0.0031
Feb9 T1	0.014	1.557	0.0571

Table 9: Dependence of spurt loss on FDP-S875 concentration

Figure 34 shows three tests conducted with the dehydrated FDP-S875 product ground in the cyclone mill by using the 0.25mm screen. As before the test conditions are 200°F and 300 psi, further the KCl concentration in the fluid system has been reduced from 16.8 to 6% KCl. Below graphs confirm that decrease in FDP-S875 concentration is not generally linked to increased spurt loss. The spurt loss obtained at a concentration of 0.015g/ml is similar to the spurt loss from previous tests with the coarser product at the same concentration. Decreasing the concentration by 0.0025g/ml resulted in a decrease of spurt loss from 0.85 to 0.21gal/ft² (yellow graph). Again this result makes it unlikely to be able to predict spurt loss via adjustment of fluid loss control additive concentration. Further reduction in S875 concentration to 0.01g/ml did not create a sealing filter cake on the sand disc surface.

Figure 35 shows two of the filter cakes referred to in the graphs of Figure 34. In general the filter cakes look similar to those created with the coarser version of FDP-S875. The filter cake is non-uniform and in the cakes created with 6g and 5g S875 it can be seen how the soft particles stick together and lay down on the disc surface this way. Lying down of clumps instead of single particles lead to faster shut off of fluid flow and results in low spurt loss values. These low spurt loss values can be confirmed by the tests done so far. The filter cake on the left hand side seems to be the most uniform. The difference to the other three cakes shown is that it is created at 100psi instead of 300psi. Even though the amount of S875 varies throughout the displayed filter cakes no difference can be seen in the amount of product deposited on the disc. Indeed when the sand disc is removed from the HPHT cell and the brine which remained in the cell body is poured out it can be seen that FDP-S875 is still in the brine. Not the entire amount of added fluid loss control additive is needed to seal off the filter disc surface. This leads to the conclusion that despite the fact that the lowest concentration tried within this testing series failed to seal it might be possible to reduce the S875 concentration.

From the hydration tests described before it is known that at about 200°F the particles soften and stick together immediately. As the set up of the stirring device takes about 2 minutes after the brine has been poured in the HPHT cell body the particles had already formed clumps when the stir was switched on. From the filter cake textures and the low spurt loss values it can not be said for sure that the stirring allows single particles to settle down. Even if bigger clumps are destroyed by the motion of the stir smaller particle accumulations may still exist. At a temperature this high not only the outer part of the grains are soft, the complete grain is swollen, soft, totally hydrated and therefore cross linked to other particles.

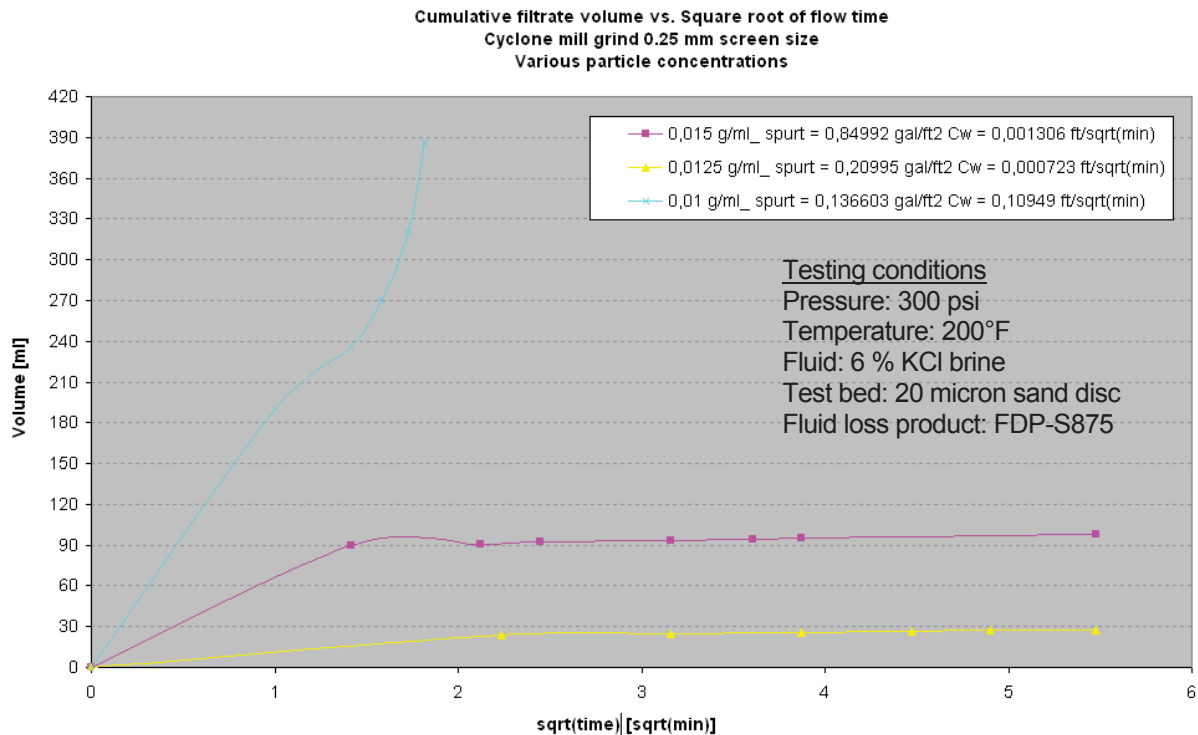


Figure 34: Dependence of spurt loss on ground FDP-S875 concentration



Figure 35: FDP-S875 on 20 micron sand discs

3.3.2.1 Adding Surfactants to the fluid system

As it has been described in chapter 3.4.2 the soft particles clump together and therefore their settlement on the disc surface occurs pretty quickly and the resulting filter cake is non-uniform and prone to breakthroughs.

Therefore it has been tried to add surfactants to the fluid system in order to prevent the premature swelling, softening and stickiness of the particles. The surfactants added are Rhodasurf BC-420 and Losurf 2000L. Rhodasurf BC-420 is a clear viscous liquid with slight odor. According to its material safety data sheet (MSDS) it is slightly soluble in water and has a density of 0.93 to 0.938 g/ml (at 22°C). The chemical name or synonym for this product is Tridecyl Alcohol Ethoxylated. Losurf 2000L is a clear yellow liquid with mild odor and a specific gravity of 1.126 at 20°C. The material safety data sheet of the surfactant states that Losurf 2000L consists of 1-5% Sodium Sulfate and 30-60% Sulfate.

Usually Rhodasurf BC-420 is used at a concentration of 2ml per liter fluid. For the HPHT fluid loss test 400ml of 6% KCl brine were used and therefore 0.8ml Rhodasurf BC-420 was added. The HPHT cell temperature was adjusted to 200°F and a pressure of 300 psi was applied during the procedure. The fluid loss was recorded for 13.5 minutes and the data is plotted in Figure 36. For this test the finest cyclone mill grind was used resulting in an average particle size of about 105 micron. The spurt loss of this test is 111.59 cm³ which gives a spurt loss per unit area of 1.09gal/ft². With the slope of the added trendline which is 3.1431cm³/sqrt(min) a C_w value of 0.00205 ft/sqrt(time) can be calculated.

Figure 37 shows the filter cake produced with 6g of FDP-S875 on a 20 micron filter disc. The filter cake looks whiter and less clear than previously created filter cakes. Comparing this cake to the later described filter cakes created at 115°F it is non-uniform and it appears that S875 particles stick together and have been deposited like that on the filter disc.

By looking at the filter cake created by adding Rhodasurf BC-420 it seems possible that this filter cake will allow fluid breakthrough. The darker spots on the filter cake indicate that less fluid loss additive has been deposited and by applying pressure a leak would most likely occur there.

The second test using a surfactant has been conducted with Losurf 2000L. The test conditions stayed the same as in the test using Rhodasurf BC-420 as a surfactant. Again 0.8ml of surfactant has been added to the fluid system. It was not possible to create a sealing filter cake within this trial. No data points have been recorded because fluid outflow did not slow down and so at 300 psi it only takes about 20 seconds to filter 400ml brine through a 20 micron core. The resulting filter cake is shown in Figure 38. The FDP-S875 has been deposited basically in the center of the filter disc. The fluid could pass outside the center and therefore no shut off of fluid flow could be achieved. Again it becomes comprehensible that the particles kind of stick together at this temperature instead of staying evenly distributed in the stirred fluid system.

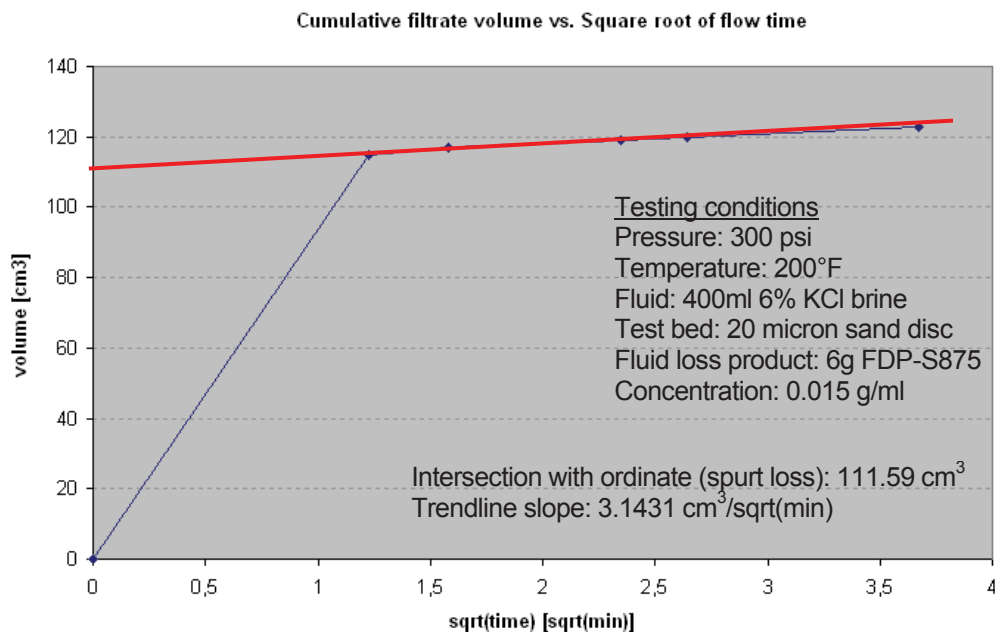


Figure 36: Surfactant Rhodasurf BC-420 fluid loss over sqrt(time)



Figure 37: Rhodasurf BC-420



Figure 38: Losurf 2000L

3.3.3 HPHT fluid loss tests at 150°F

The first test at reduced temperature was performed at 150°F. With initial fluid losses before filter cake built up of only 51ml the calculated spurt value resulted in 0.487gal/ft². The particle concentration used was 0.02g/ml and a pressure of 300psi was applied to the system. Like in previous testing at 200°F again only part of the 8 grams S875 was deposited on the disc surface. Figure 39 shows the filter cake produced at 150°F. For comparison a filter cake created at 200°F is displayed on the right. The filter cake on the left is more uniform, the one on the right hand side creates the impression of small particle accumulations within the actual filter cake. For the test at 150°F the 0.25mm screen size cyclone mill ground product has been used and the pH value of the 6% KCl brine has not been increased. The evaluation of fluid loss control ability over time led to a C_w value of 0.000547ft/sqrt(min) which indicates very good fluid loss control. Despite the good sealing ability at 150°F the spurt values are still too low to achieve a large consolidation radius around the wellbore. The defined objective is to reach at least spurt loss values per unit area of about 3gal/ft². As the current results are below this target value further reduction in temperature will be the next step.



Figure 39: 8g FDPS-875 at 150°F after 16minutes



Figure 40: 6.6g FDPS-875 at 200°F after 16minutes

3.3.4 HPHT fluid loss tests at 135°F

For the following tests the HPHT cell temperature has been adjusted to 135°F. The other testing conditions remained the same as before when testing at 150°F, more information on the testing conditions can be found in the diagrams below. Figure 41 displays the results of the first testing series at 135°F. The first thing recognized is the established trend. Except test Mar5_T3 all other tests show similar values for spurt loss as well as for the wall building fluid loss coefficient C_w . Test Mar6_T3 has been stopped right after the spurt loss in order to compare the filter cake texture with those being in the cell body for about an hour. But it can be seen that also this tests shows about the same initial losses than the others being part of this trend.

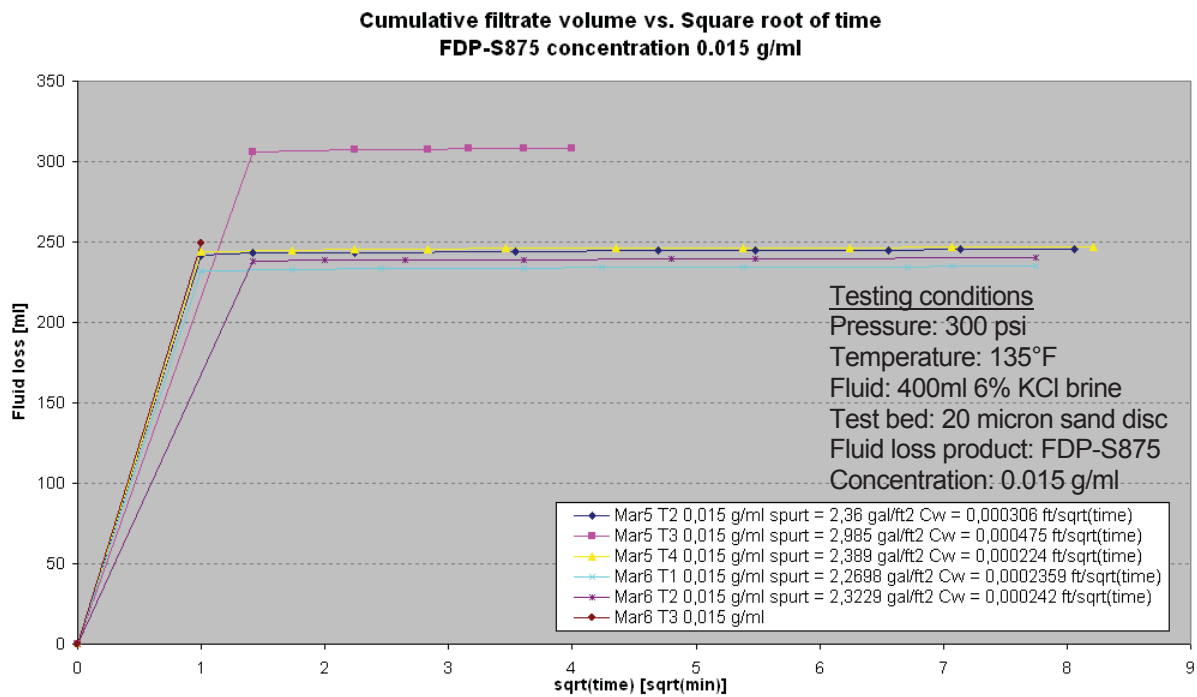


Figure 41: Fluid loss control over sqrt(time) at 135°F

In Table 10 the results of the testing performed at a FDP-S875 concentration of 0.015g/ml are summarized. For the first time the calculated spurt loss reaches promising values. As four spurt loss values of around 2.3gal/ft² confirm there is a trend created for the spurt loss achievable at a FDP-S875 concentration of 0.015g/ml at 135°F. The exceptional higher spurt value of test Mar5_T3 gives reason to expect even higher spurt losses in further tests. Also the values of the calculated wall-building fluid loss coefficient C_w are extraordinary with values around 0.0002 to 0.0004 ft/sqrt(min).

Test	FDP-S875 concentration [g/ml]	spurt [gal/ft ²]	C_w [ft/sqrt(min)]
Mar5 T2	0.015	2.36	0.00031
Mar5 T3	0.015	2.985	0.00048
Mar5 T2	0.015	2.389	0.00022
Mar6 T1	0.015	2.27	0.00024
Mar6 T2	0.015	2.323	0.00024
Mar6 T6	0.0075	1.924	0.00029
Mar6 T7	0.0075	1.718	0.00019
Mar9 T1	0.0075	1.667	0.00026
Mar9 T2	0.0075	2.01	0.00022

Table 10: Fluid loss control over sqrt(time) of various FDP-S875 concentrations (135°F)

Above table further includes the results obtained from testing after the S875 concentration has been reduced to 0.0075g/ml. Figure 42 shows the results of the testing at reduced concentration compared to the HPHT tests done before using 0.015 grams of S875 per milliliter. Similar to some results described in chapter 3.4.2 the reduction in particle concentration did not lead to increased spurt loss as it might be expected and as it is desired. Actually the cumulative filtrate volume of these tests was below those performed at higher S875 concentration. The graphs are marked by a red circle in the diagram below. It can be seen that within the four tests two trends were created. One trend resulting in spurt values of about 2gal/ft² (test Mar6_T6 and Mar9_T2) and the other trend giving spurt values of about 1.7gal/ft² (test Mar6_T7 and Mar9_T2). Detailed values of spurt loss and C_w can be looked up in Table 10.

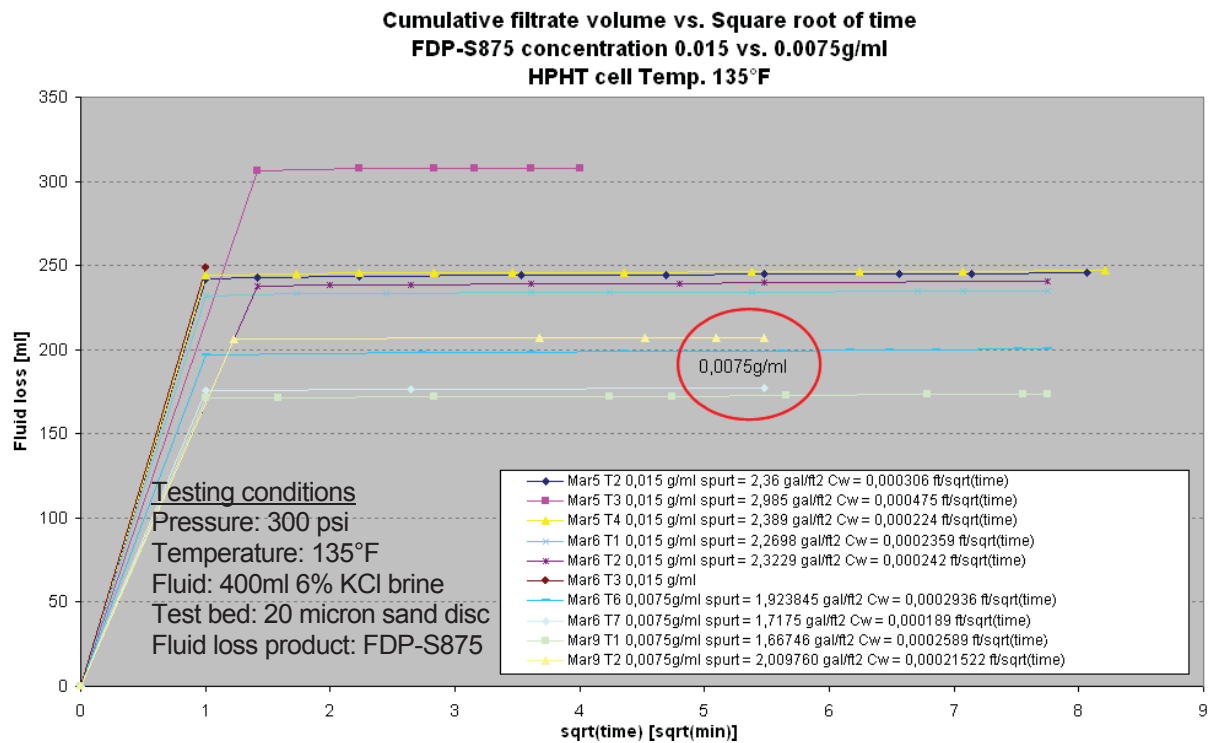


Figure 42: Fluid loss control over sqrt(time) of various FDP-S875 concentrations (135°F)

When looking at the pictures taken of two of the created filter cakes the first thing noticed is that the filter cake is really thick. Although the product used has not been increased but reduced compared to the test done at 150°F the filter cake is much thicker than before. For the first time the entire amount of S875 has been deposited on the sand disc and has been actually used for fluid loss control. The texture of the cake shown in Figure 43 is similar to those created at the filter paper after pouring water on top of the established cake. The top of the filter cake was perfectly cross linked and therefore a gel with no void space. On its bottom the filter cake is hard and not flexible. The filter cake on the left side has been in the testing apparatus for one hour the top of the cake is kind of round the gel has expanded in the fluid. The filter cake on the right has been taken out of the HPHT cell right after spurt loss had been finished and the filter cake had been built up. The entire texture of this cake resembled the bottom part of the first one, it was a stiff gel.

From this testing series it can be concluded that it has to be possible to further reduce the concentration of S875. At lower temperature the filter cake creation seems to be delayed due to the deposition of single particle grains instead of entire soft grain accumulation which will cover a surface area faster and results in low spurt loss values. Further it has to be analysed if the reduction in product concentration leads to decrease in spurt loss also at lower temperature and lower product concentration.



Figure 43: 6g FDPS-875 at 135°F after 1 hour (0.015g/ml)



Figure 44: 6g FDPS-875 at 135°F after 2 minutes (0.015g/ml)

3.3.5 HPHT fluid loss tests at 115°F

The first test at 115°F was dedicated to compare the performance of 0.015g/ml FDP-S875 in 135°F brine with its performance in 115°F brine. The created filter cake can be seen in Figure 45, the cake was brittle and the particles had not been cross linked yet. This is why the cake broke by taking it out of the HPHT cell. The only difference between the filter cake shown in Figure 44 and the cake created now is the lower temperature. At 115°F the used 6g S875 in 400ml 6% KCl brine failed to establish a seal. Temperature and pressure conditions (still 300psi) seemed to be too low to form a gel filter cake when using 6g of S875. For the next test the fluid loss control additive concentration has been reduced to 0.0075g/ml (which means 3g in 400ml brine). The resulting filter cake is shown in Figure 46. It is a beautiful uniform filter cake with the entire amount of added S875 deposited on the sand disc surface. The filter cake still shows potential to reduce the amount of S875. This assumption was made because when testing on filter papers the concentration was reduced so much that only a S875 film as thin as the filter paper itself was able to seal off the paper. Of course in the HPHT cell the pressure conditions are a lot higher but the assumption was to be able to further reduce the S875 concentration. The question arising from the first two tests is why the higher S875 concentration failed to seal off the porous disc. Has it been one test which simply failed or can this phenomenon be connected to the fact that at 135°F the lower FDP-S875 concentration sealed off the filter disc faster and resulted in less cumulative losses and lower spurt values.



Figure 45: 6g FDPS-875 at 135°F (0.015g/ml) – no seal created



Figure 46: 3g FDPS-875 at 135°F (0.0075g/ml)

Further the picture of this brittle filter cake raises the question if the particles are deposited in this grain like texture and if the packing reduces fluid throughput to a degree that the warm brine on top

of the cake leads to cross linking of the particles which leads to creation of a seal. What speaks against this theory is the fact that the seal is established right away after spurt loss. As it can be seen in the graphs there is no spurt loss followed by reduced fluid throughput. There is basically total seal after spurt loss. The high spurt loss and the subsequent immediate seal (which can be seen also in the following graphs presented in this chapter) leads to another theory.

What might happen is that at lower temperature the FDP-S875 particles do not soften and swell entirely in the brine. Particles which have a hard core and a soft outer boundary might be created. In this case the grains start to hydrate and they already have a soft coating. On the other hand in this stage they do not stick together yet and keep suspended as single particle grains while stirring the fluid system. When pressure is applied and the discharge valve is opened the particles do not settle as already cross linked particle clumps covering large parts of the filtering area. By applying pressure and opening the discharge valve the suspended particles settle down and pressure, temperature and time allow the particles to cross link. As the particle size of the used material ranges from about 10.8 to 300 micron it is also possible that the larger particles become a soft coating and remain hard in the center. These larger particles will settle on the sand disc first. Fluid flow will still occur until smaller maybe entirely hydrated particles settle down and close the void spaced.

What happens exactly is room for further studies and analysis. But the idea and performance of a particle which has a hard core and is soft on the outside may lead to further projects within Halliburton.

The temperature at which this theory works best has been 115°F. In the following variations in particle concentration and their results are plotted and described.

Above time is mentioned beside temperature and pressure as one of the factors influencing the cross linking of FDP-S875. During the tests at 115°F the brine has been heated in the HPHT cell body. At temperature the S875 has been added and has been mixed in the brine by stirring shortly by hand. Immediately the set up of the stirring device and pressure inlet started. This procedure takes about 2 minutes. No more time passed before the pressure had been applied and the discharge valve was opened. This is mentioned because later on it has been tested if this soaking time of S875 in 115°F warm brine can be extended without interfering with the great results of spurt loss and seal.

In below four images filter cakes created at 115°F are shown. The filter cake in Figure 47 is created with 1g of FDP-S875 which means the concentration has been reduced to 0.0025g/ml. The filter cake is thin and uniform. The texture does not show signs of premature particle accumulations settled as clumps on the disc surface. The next three pictures show a filter cake created with 0.6g of FDP-S875. It has to be mentioned that out of several trials only two filter cakes could be created with 0.6g of product. Using 0.75g of FDP-S875 which means a concentration of 0.001875g/ml in 400ml brine the filter cake built up has been successful throughout the entire testing at 115°F.

As the figures show how thin the cakes are it can be concluded that about 0.75g of S-875 are needed to be sure to cover the filtration area of the disc (3.892in^2 or 25.12cm^2). This leads to the assumption that for sealing off a porous medium under a pressure of 300psi at least 0.193g/in^2 respectively 0.0299g/cm^2 of FDP-S875 is needed. The expression porous medium is used in the previous sentence because the values do not only refer to the 20 micron sand disc. Later it will be seen that it is true also for the 5, 10 and 35 micron sand discs (the values indicate the average pore diameter of the sand discs).



Figure 47: 1g FDPS-875 at 115°F (0.0025g/ml)



Figure 48: 0.6g FDPS-875 at 115°F (0.0015g/ml)



Figure 49: 0.6g FDPS-875 at 115°F (0.0015g/ml)



Figure 50: 0.6g FDPS-875 at 115°F (0.0015g/ml)

**Cumulative filtrate volume vs. Square root of time
FDP-S875 concentration 0.0075 vs. 0.005g/ml
HPHT cell Temp. 115°F**

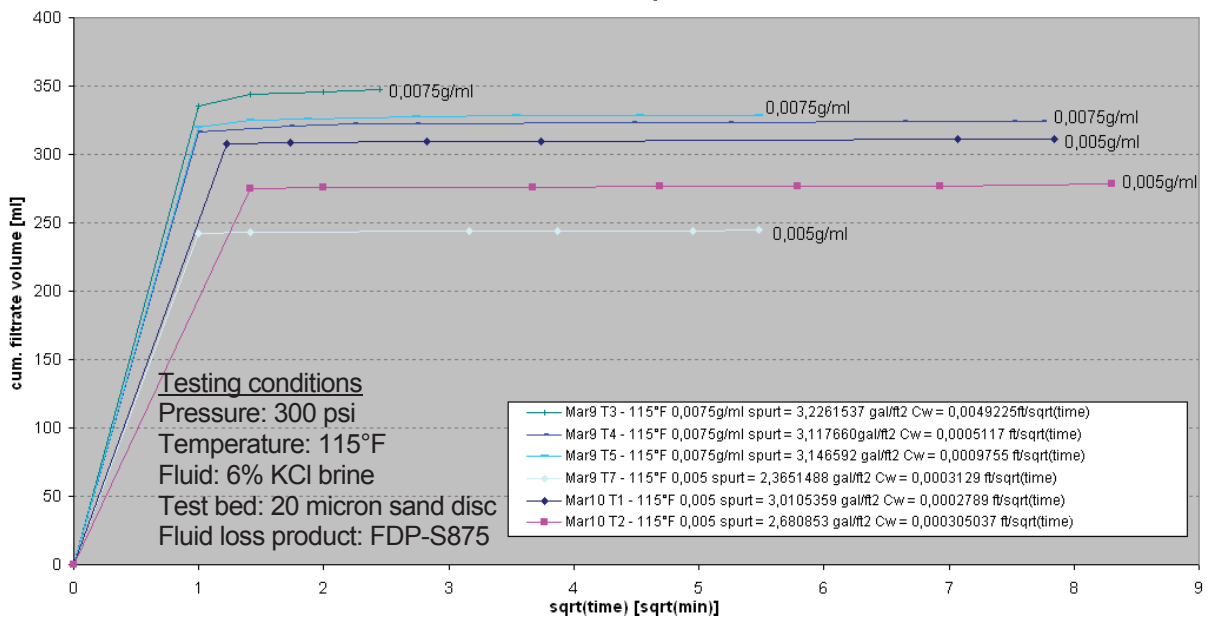


Figure 51: Comparison of fluid loss control over sqrt(time) (0.0075 vs. 0.005g/ml)

Above Figure shows six fluid loss tests at two different FDP-S875 concentrations. BY looking at the graph it can be seen that the same phenomenon as in previous tests at 135°F occurs. The higher concentration 0.0075g/ml leads to higher cumulative fluid loss and higher spurt values as the lower concentration (0.005g/ml). As Table 11 shows, the spurt values achieved with 0.0075 grams S875

per milliliter are above 3.1gal/ft². At a S875 concentration of 0.005g/ml the highest obtained spurt value lies at 3.01gal/ft². The other two results at the same conditions show spurt values of only 2.37 and 2.68gal/ft². Concerning C_w it can be stated that with one exception the values are below 0.0009 ft/sqrt(time) which means that the wall building ability of the product is very good.

The next diagram (Figure 52) compares a FDP-S875 concentration of 0.005 and 0.0025g/ml. For the first time it can be seen that lowering the product concentration actually resulted in an increase in spurt loss. Unfortunately it can be seen that the results do not show a trend of lower concentration resulting in higher spurt loss. Just two (Mar10_T4 and Mar10_T5) out of six tests with 0.0025g/ml S875 showed higher spurt loss than the higher concentration (0.005g/ml). Three tests on the other hand showed that the lower S875 concentration can lead to less cumulative fluid loss and lower spurt values. It is promising that spurt values as high as 3.36gal/ft² could be obtained and potential for further reduction of concentration can be seen. But again the randomness in particle settlement and filter cake build up do not allow making accurate predictions of spurt values with variation of fluid loss control additive concentration.

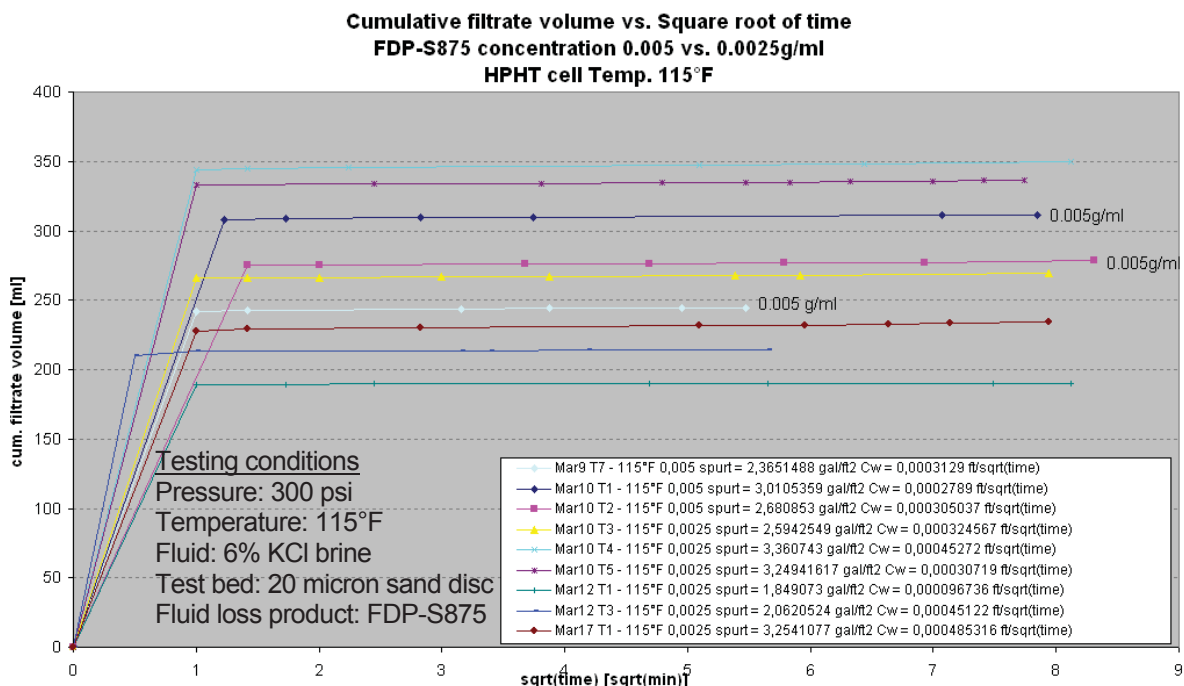


Figure 52: Comparison of fluid loss control over sqrt(time) (0.005 vs. 0.0035g/ml)

In the diagram below the comparison between a FDP-S875 concentration of 0.0025 and 0.001875g/ml is shown. This diagram displays the trend that the higher concentration (0.0025g/ml is marked on the graph) basically gives the lower spurt values and the cumulative filtrate volume lies below those of the lower concentration. Just two exceptionally high spurt values of the tests using 0.0025g/ml S875 lie above some of those at lower concentration. But three tests using the reduced particle concentration could top the results of the previous tests. Mar11_T1 reached a spurt value as high as 3.66gal/ft². Assuming a formation porosity of 30% and a wellbore with a diameter of 5 inch this spurt value would lead to a reservoir penetration depth of about 0.64ft. An overview of all spurt and C_w values obtained from testing at 115°F can be found in Table 11.

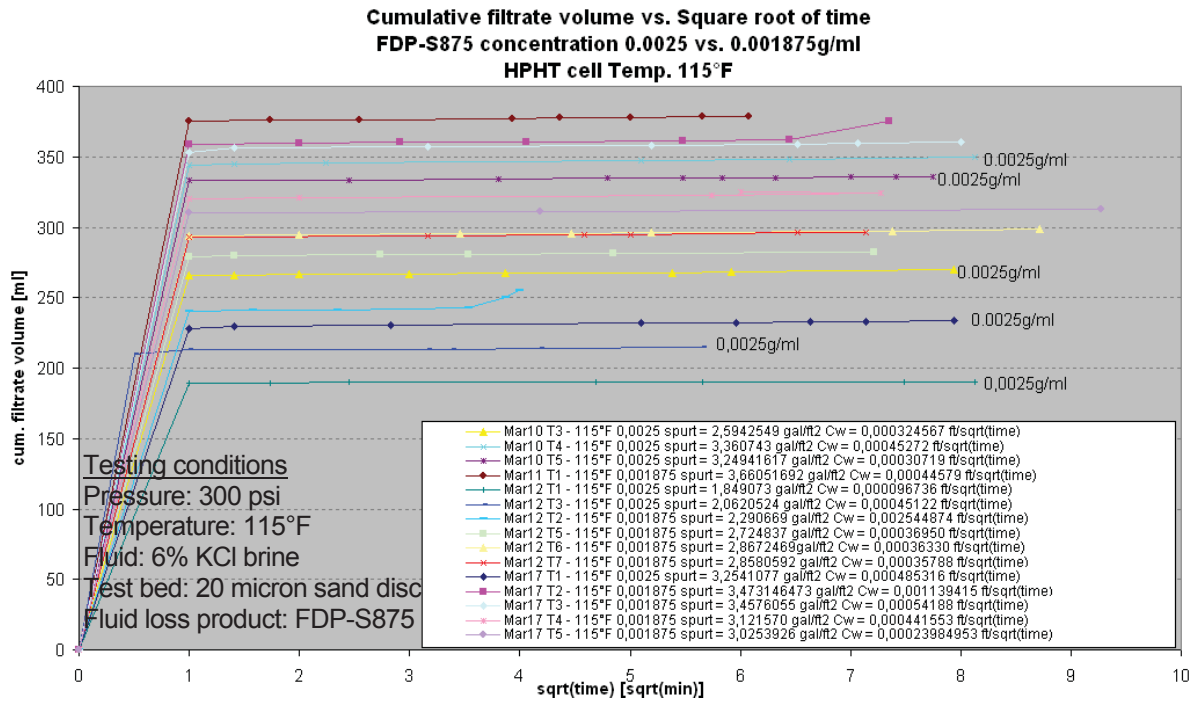


Figure 53: Comparison of fluid loss control over sqrt(time) (0.0025 vs. 0.001875g/ml)

As mentioned previously it has been tried to reduce the concentration of S875 to 0.0015g/ml. It just worked twice; the results can be seen in the diagram below. The two graphs showing the fluid loss recorded at a S875 concentration of 0.0015g/ml are marked. It can be seen that the reduction did not lead to increased spurt loss. As reducing the concentration did not lead to the desired result but makes it unsure if a sealing filter cake can be created it is recommended to use a FDP-S875 concentration of 0.001875g/ml for further testing which will analyse the influence of formation permeability on spurt loss and sealing ability of this product.

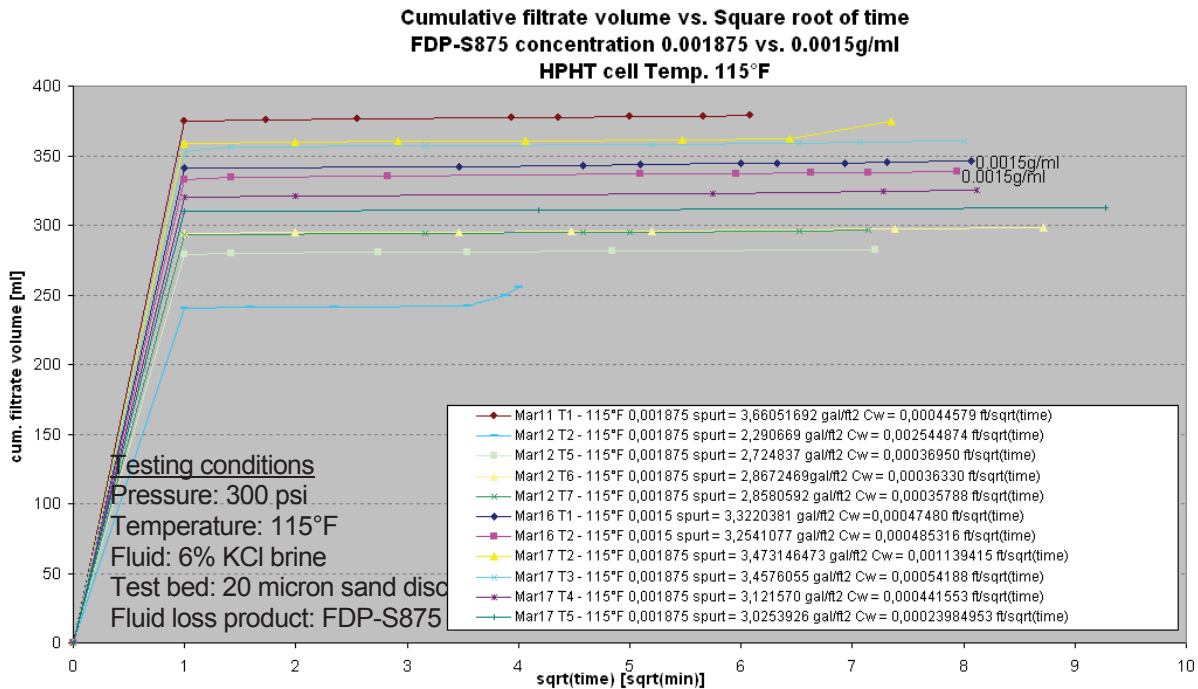


Figure 54: Comparison of fluid loss control over sqrt(time) (0.001875 vs. 0.0015g/ml)

Test	average pore diameter of disc [micron]	temperature [°F]	pressure [psi]	concentration [g/ml]	spurt loss [gal/ft ²]	C _w [ft/sqrt(time)]
Mar9 T3	20	115	300	0.0075	3.2261537	0.0049225
Mar9 T5	20	115	300	0.0075	3.146592	0.0009755
Mar9 T4	20	115	300	0.0075	3.11766	0.0005117
Mar10 T1	20	115	300	0.005	3.0105359	0.0002789
Mar10 T2	20	115	300	0.005	2.680853	0.000305037
Mar9 T7	20	115	300	0.005	2.3651488	0.0003129
Mar10 T4	20	115	300	0.0025	3.360743	0.00045272
Mar17 T1	20	115	300	0.0025	3.2541077	0.000485316
Mar10 T5	20	115	300	0.0025	3.24941617	0.00030719
Mar10 T3	20	115	300	0.0025	2.5942549	0.000324567
Mar12 T3	20	115	300	0.0025	2.0620524	0.00045122
Mar12 T1	20	115	300	0.0025	1.849073	0.000096736
Mar11 T1	20	115	300	0.001875	3.66051692	0.00044579
Mar17 T2	20	115	300	0.001875	3.473146473	0.001139415
Mar17 T3	20	115	300	0.001875	3.4576055	0.00054188
Mar17 T4	20	115	300	0.001875	3.12157	0.000441553
Mar17 T5	20	115	300	0.001875	3.0253926	0.00023985
Mar12 T6	20	115	300	0.001875	2.8672469	0.0003633
Mar12 T7	20	115	300	0.001875	2.8580592	0.00035788
Mar12 T5	20	115	300	0.001875	2.724837	0.0003695
Mar12 T2	20	115	300	0.001875	2.290669	0.002544874
Mar16 T1	20	115	300	0.0015	3.3220381	0.0004748
Mar16 T2	20	115	300	0.0015	3.2541077	0.000485316

Table 11: Summary of tests on 20 micron discs at 115°F

By comparing the particle concentration of the tests performed at 200°F with the particle concentration at 115°F a huge reduction can be determined. While at 200°F some tests already failed at a S875 concentration of about 0.014g/ml the concentration could be reduced down to 0.00187g/ml. As mentioned two tests even worked out at a concentration of 0.0015g/ml, but to be on the safe side it is recommended to go for the 0.001875g/ml. During the entire testing series at 115°F the sealing ability of the product was extraordinary. It is remarkable that although most of the tests lasted for about an hour no fluid breakthroughs occurred at this temperature. The explanation might be the extreme uniform texture of the filter cakes. The S875 particle clumps occurring at higher temperatures made the previously created filter cakes less reliable than those created at 115°F.

Finally the difference in spurt loss created by temperature is shown in Figure 55 and Table 12. All seven tests were performed with a S875 concentration of 0.0075g/ml in 6% KCl brine and at a pressure of 300psi. The diagram shows clearly that at 115°F more brine is filtered through the sand disc. At both temperatures the cumulative filtrated volume is basically lost as spurt loss before the build up of the filter cake. After the particles are deposited on the sand disc the fluid throughput is reduced to a minimum. Although the tests at 115°F still show very good C_w values those obtained at 135°F are better (Table 12). This slightly worse performance in sealing ability can be accepted by keeping in mind that the increase in spurt values ranges from 1.137 to 1.559gal/ft². It has to be mentioned that just these incremental spurt losses are much higher than the spurt values created at 200 and 150°F.

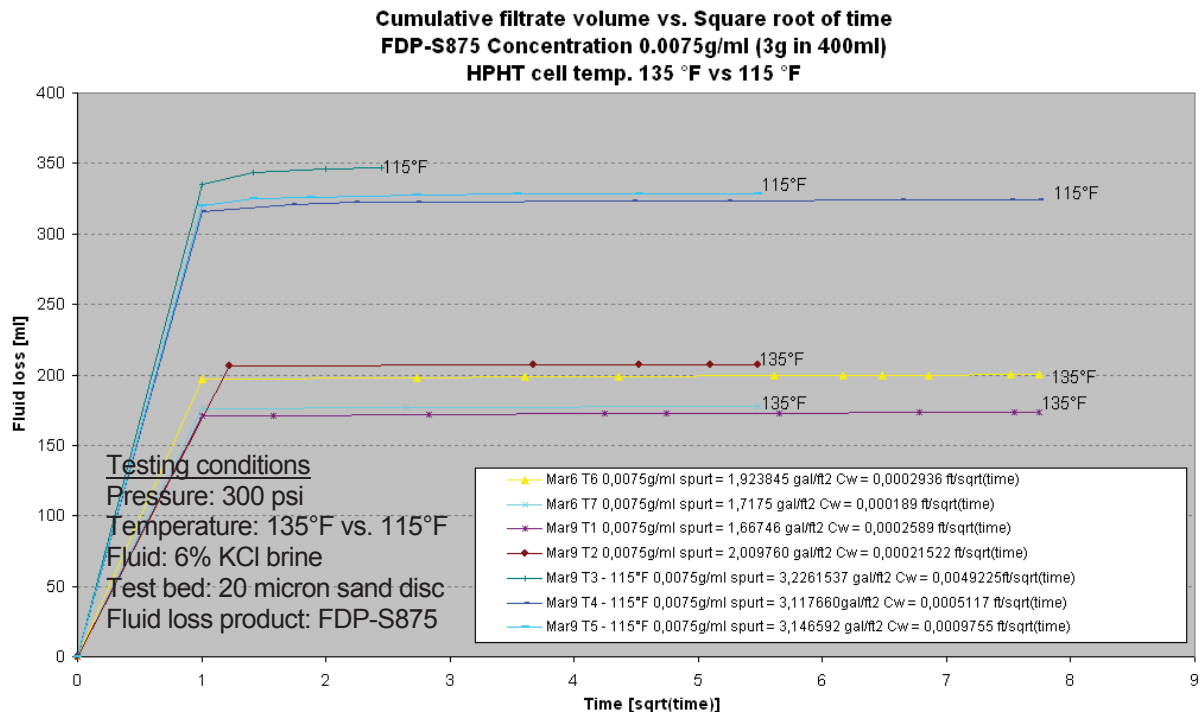


Figure 55: Temperature dependence of spurt loss and C_w

Test	FDP-S875 concentration [g/ml]	Temperature [°F]	spurt [gal/ft ²]	C _w [ft/sqrt(min)]
Mar6 T6	0.0075	135°F	1.924	0.00029
Mar6 T7	0.0075	135°F	1.718	0.00019
Mar9 T1	0.0075	135°F	1.667	0.00026
Mar9 T2	0.0075	135°F	2.01	0.00022
Mar9 T3	0.0075	115°F	3.226	0.00492
Mar9 T4	0.0075	115°F	3.118	0.00051
Mar9 T5	0.0075	115°F	3.147	0.00098

Table 12: Temperature dependence of spurt loss and C_w

As this testing series is just the beginning of the testing FDP-S875 the product testing has to be switched to other devices in the future. So far it has been proved that S875 is able to create a high spurt loss and very good fluid loss control over time. Within this thesis it will also be tested how spurt loss and sealing ability are influenced by variation in permeability. These tests can still be conducted in the HPHT cell but as soon as it comes to test the effectiveness of diversion a Hassler device has to be used. With this device it is possible to test three cores of various permeabilities and find get an idea of the actual diversion suitability of S875. For the Hassler device bigger fluid volumes are needed and it takes longer to set up the apparatus. Therefore it is necessary to know the effect of FDP-S875 soaking time in brine on its fluid loss control abilities. It has been figured out that everything works fine when applying pressure and open the discharge valve after about 2 minutes. But what if the product has to stay in the heated brine longer? To get an idea of the products behaviour by varying the soaking time in brine the following tests have been done.

3.3.5.1 Variation of FDP-S875 soaking time

It has already been explained that it has to be tested how the performance of FDP-S875 depends on the soaking time of the product in 115°F 6%KCl brine. This chapter summarizes the results of these tests. Figure 56 shows the three sand discs used for these tests. First it has been tried to pour the S875 in the HPHT cell and stir it for 40 minutes. After 40 minutes 300psi pressure has been applied and the discharge valve was opened. The result was no fluid loss control at all. The reason could be seen after removing the disc from the cell body. No fluid loss control additive has

been deposited on the disc respectively remained on the disc. Therefore it has been decided to reduce the soaking time to 15 minutes. The result was basically the same than before, no fluid loss control and literally one grain of FDP-S875 remained in the filter disc. Further reduction in product soaking time to 5 minutes resulted in the filter cake on the right. The product had already been hydrated and remained on the filter disc but no seal could be created. These results lead to the conclusion that the ideal soaking time is defined very narrow. Five minutes of soaking time seem already to impair the creation of a sealing filter cake.



Figure 56: Various soaking times of 0.75g FDP-S875 in 115°F 6%KCl brine

3.3.6 HPHT fluid loss tests at room temperature

Further it has been tried to reduce the temperature to 80°F (above RT) and the soaking time below two minutes. No sealing filter cake could be created this way. Figure 57 shows the texture of the deposited FDP-S875 particles. The particles are still white and not hydrated and soft, the particles are stuck to the core on the outer boundary but in the center it was brittle and the product could be removed easily. The next test has been performed at actual room temperature (70°F) and the particles were allowed to soak in the brine for 34 minutes because according to the previously conducted hydration tests this is the time frame after which the particles start to swell at RT.



Figure 57: 1g FDP-S875 2min soaking time in 80°F brine



Figure 58: 1g FDP-S875 34min soaking time RT brine

Also the second test failed to establish a sealing filter cake. Figure 58 shows that the particles are hydrated but it seems as if they stayed in the brine too long because the particle texture is similar to those soaking for 5 minutes at 115°F (Figure 56 rightmost filter cake). Therefore the next step was to reduce the soaking time at room temperature to 15 minutes. Test Apr1_T1 showed that a soaking time of five minutes at room temperature will successfully create a sealing filter cake. In order to confirm the result the test has been repeated (Apr1_T2) and again showed a good filter cake build up. The one thing confirmed by the second test is the sealing ability; otherwise the pink graph shows higher cumulative fluid loss as well as higher spurt loss. Test Apr1_T1 has a spurt loss of 1.72gal/ft² and its C_w is 0.0034ft/sqrt(min). Test Apr1_T2 shows a spurt value of 2.65gal/ft² and a C_w of 0.0085ft/sqrt(min). Compared to the values of C_w from testing at 115°F the numbers at RT are worse.

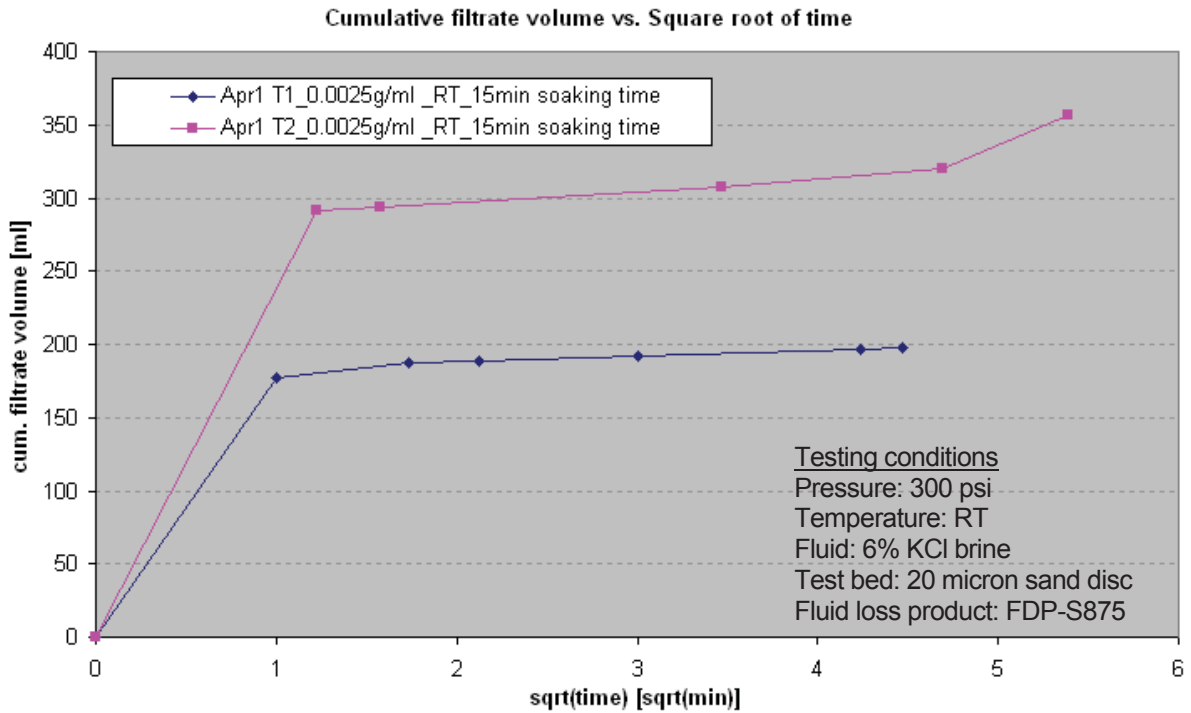


Figure 59: HPHT test at RT – 15 minutes FDP-S875 soaking time



Figure 60: 1g FDP-S875 15min soaking time RT brine

The created filter cakes looked as shown in Figure 60. At first sight it looks clear and similar to the cakes created at 115°F. By taking a closer look it can be seen that some grains are still visible, especially in the center of the filter cake. Touching the filter cake leads to the observation that it is not that soft and flexible as the cakes created at elevated temperature. Testing showed that at room temperature longer soaking time is possible without resulting in failure to seal off the disc.

3.3.7 FDP-S875 filter cake invasion study

As the filter cakes created at 115°F showed very promising results concerning initial fluid loss per unit area as well as extraordinary good sealing ability over time one filter cake has been handed in for invasion analysis. The following images and data have been created by the analytical department of the Halliburton Technology Center during an invasion study using a scanning electron microscope (SEM).

The filter cake handed in for analysis was created with 1g of FDP-S875 on a 20 micron filter disc. In 400ml of 6% KCl brine the addition of 1g fluid loss additive results in a product concentration of 0.0025 g/ml. Before performing the SEM work the filter cake sample has been oven dried. In order to get a better idea of the measurements in the following description of the results it has to be mentioned that the thickness of the sand disc is ¼" or 6350 micrometer.

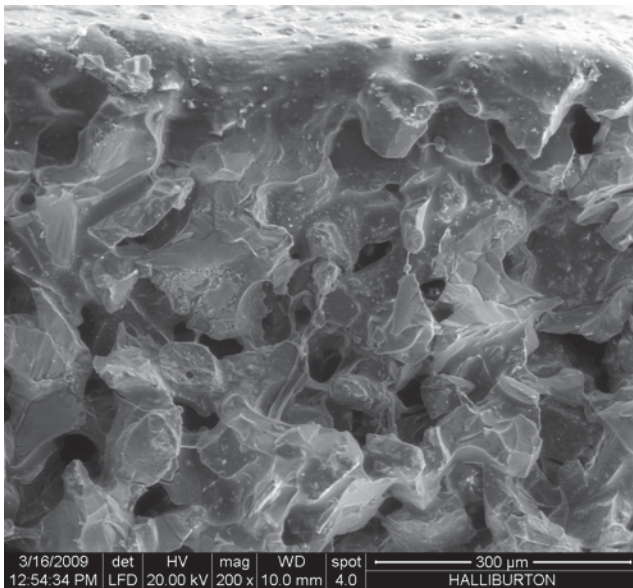


Figure 61: Top edge of the guar filter cake

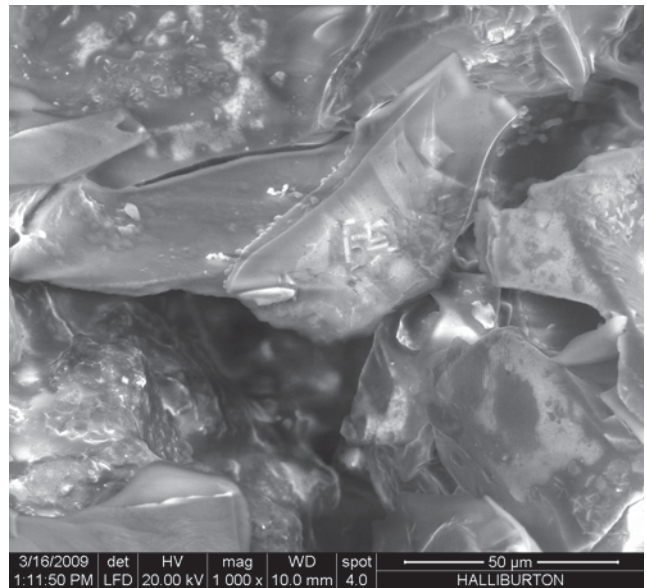


Figure 62: Detail of the core at 300 microns from the surface

According to the report submitted by the analytical department the guar material can be found primarily in the top 150 microns of the sand disc. Figure 61 shows the top of the sand disc covered by the FDP-S875 filter cake at a 200x magnification. Further the invasion of the guar into the top 150 to 200 microns of the sand disc can be seen. At a depth of 200 micron only low guar concentration could be found. No guar could be detected deeper in the sand disc and although some particles might be present no impairment of porosity occurs. Figure 62 shows an image of the core disc at 1000x magnification. The center of the image is 300 micron below the sand disc surface. By comparing this image to Figure 64, which is a 1000-fold magnification of the core at 100 micron below surface, it can be seen that no guar particles are present. At a depth of 100 micron on the other hand the guar particles are clearly visible in the pore space. For comparison an image 1000 micron from the disc surface has been added (Figure 63). Again it shows the porosity of the 20 micron filter disc unimpaired by guar particles.

As the fluid loss additive was detected up to 200 microns below the filter disc surface it can be stated that only about one thirtieth of the disc thickness are invaded by the guar. To create a thin and minimal invasive filter cake in order to prevent formation damage and reduce the resulting skin has always been the industries objective. It was possible to reduce the amount of fluid loss product to 0.75g to cover the filter disc surface. As invasion study was performed with 1g of FDP-S875 and just at the 20 micron filter disc it is recommendable to conduct further invasion studies on the 5, 10 and 35 micron filter discs.

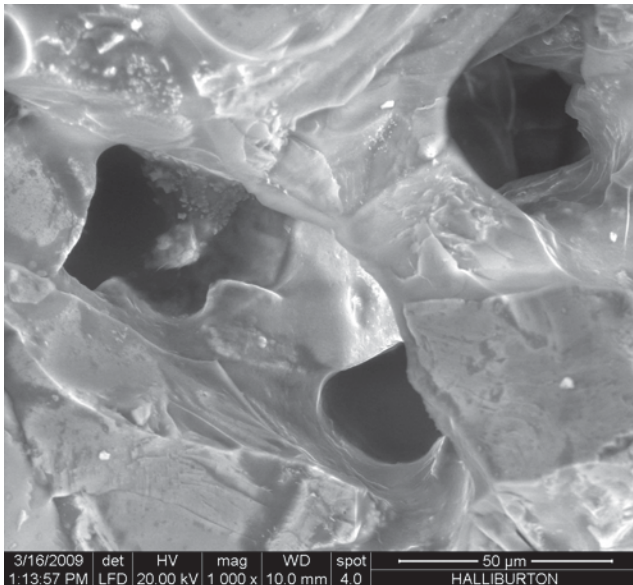


Figure 63: Detail of the core at 1000 microns from the surface

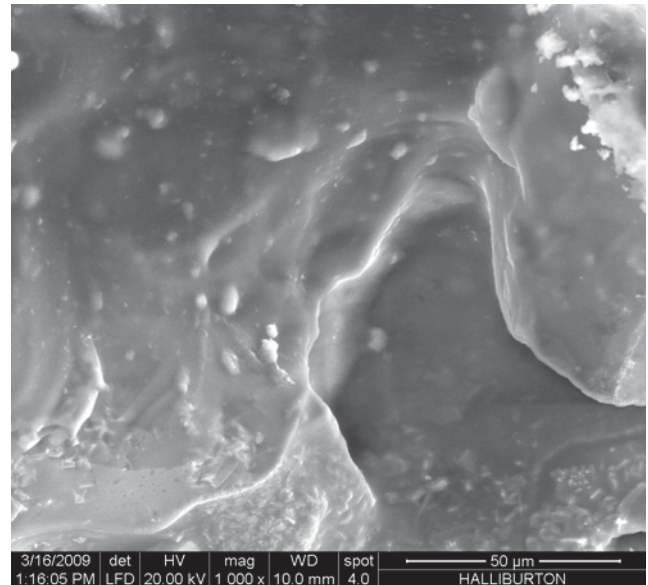


Figure 64: Detail of the core at 100 microns from the surface

3.3.8 Conclusion

The objective of the performed testing was to figure out if the developed FDP-S875 product is suitable for the diversion concept defined at the beginning of chapter 3. This means in terms of diversion that the objective is not so much to equalize permeability throughout the production interval and to achieve uniform distribution because the filter cake permeability is about the same. The objective is to manage resin injection with spurt loss and then basically seal off the zone which leads to diversion of the resin. This explains once again the need for approximately the same spurt losses on various permeabilities.

It should be analysed if the product acts at certain condition exactly the way that from an engineering point of view it would be suitable to fulfill the prerequisites defined for the concept. As mentioned before the conditions at which it works the desired way are pretty narrow. Therefore chemists may have to modify the product and give it more stability at higher temperature which would delay swelling, softening and the formation of particle clumps. One possibility might be to increase the pH value of the product. It can be concluded that this product does have the ability to combine high spurt loss with very good fluid loss control abilities. If it also fulfils the prerequisite of not being susceptible to variation in formation permeability will be seen in continuing testing.

3.4 Testing Barcarb® as a Diverting Agent

Barcarb® is a registered trademark of Halliburton and already used in the petroleum industry. Barcarb (calcium carbonate) is available in six grades 5, 25, 50, 150, 600 and 2300. According to the material safety data sheet (MSDS) it is composed of crystalline silica, quartz and limestone. Barcarb 5, 25 and 50 are used to increase the density of water- and oil-based muds as well as of brine-based fluids. Further these grades are used for fluid loss control. Barcarb 50, 150, 600, and 2300 are used as bridging agents to solve lost circulation problems and for squeeze mixes. Depending on the grade Barcarb is an odorless white powder or granule with a specific gravity of 2.7 to 2.78. The product is insoluble in water but fully soluble in 15% HCl solution. Using Barcarb as a bridging agent it is recommended to add $14.26 - 28.53 \text{ kg/m}^3$ of the appropriate Barcarb grade. To maximize bridging effectiveness the average particle size of the bridging agent should be equal or not less than one third of the formation pore diameter.^[16] The figure below shows the particle size distribution of four Barcarb grades. According to the chart below, which is taken from the Barcarb product data sheet, the number included in the product name refers approximately to

the average particle size of the product. The exception is Baracarb 5; the average particle size of this fluid loss additive is according to the chart about 10 microns.

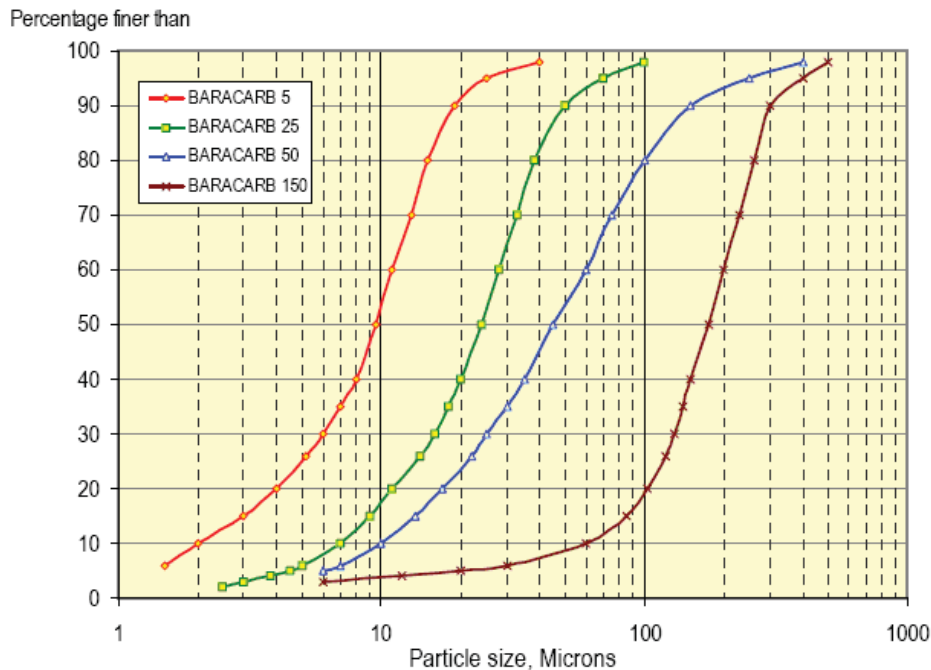


Figure 65: Baracarb agent particle size range ^[16]

The objective of the following tests is to evaluate if the concept of injecting resin via spurt loss is applicable with Baracarb as it is with FDP-S875. In order to prevent wasting of resin the determination of the spurt loss achievable was done by using 6% ACL brine.

3.4.1 Basic product behaviour

The tests to evaluate the basic product behaviour of various Baracarb grades were conducted according to the test procedure described in chapter 3.2.3. As Baracarb is insoluble in water and does not show any particle swelling the tests were performed at room temperature only. Different test beds were provided by using 11 and 25 micron Whatman filter paper. The performance of several Baracarb grades and concentrations has been tested on the filter papers.

As it was expected due to the manufacturer's recommendations of average particle size for effective bridging the Baracarb grade showing the best fluid loss control on both filter papers is Baracarb 5. One third of the pore diameter of the 11micron filter paper is 3.66 micron and one third of the pore diameter of the 25 micron filter paper is 8.33 micron. The average particle size of Baracarb 5 (10 micron) is not equal but more than slightly higher than one third of the pore diameter of both filter papers.

Without adding any fluid loss product it takes 2 respectively 5 seconds for 200ml of 6% ACL brine to be sucked through a 25 respectively 11 micron filter paper by the house vacuum line of the Halliburton Technology Center. This results in a flow rate of 100 ml/sec for the 25 micron filter paper and a flow rate of 50 ml/sec for the 11 micron filter paper. By adding the Baracarb products the results plotted below could be seen.

Figure 66 shows a comparison of the flow rate of 6% ACL brine through 11 micron filter paper by adding various Baracarb grades and concentrations.

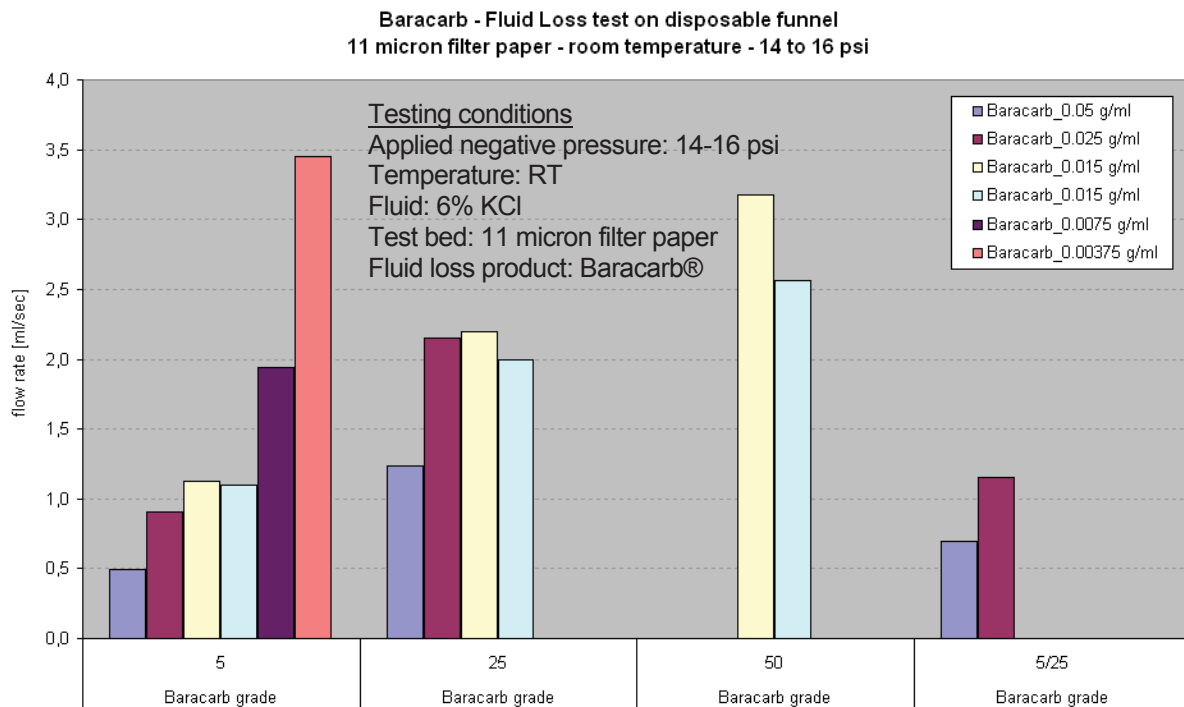


Figure 66: Baracarb fluid loss test on 11 micron filter paper

Baracarb 5 clearly shows the best fluid loss control. At the same product concentrations the flow rate could be reduced the most by using Baracarb 5. With increasing particle size the pore space in the filter cake increases and reduces bridging efficiency. The idea was to be able to control the spurt loss and therefore resin penetration depth by varying grade and concentration of Baracarb. Although the differences resulting from variation of both grade and concentration are significantly no results suitable to be used in the diversion concept described could be found.

The mixture of Baracarb 5 and 25 resulted in lower flow rates than using Baracarb 25 only. In this mixture the heavier Baracarb 25 particles will settle on the filter paper first. The smaller particles will settle afterwards to fill and reduce the remaining pore space. This result in a significant reduction in flow rate compared to the use of Baracarb 25 only.

As the tests showed even the Baracarb 5 particles are not small enough to completely bridge off the filter paper and create a seal.

11µ feilter papper	Baracarb grade 5	Baracarb grade 25	Baracarb grade 50	Baracarb grade 5/25
concentration [g/ml]	[ml/sec]	[ml/sec]	[ml/sec]	[ml/sec]
0.05	0.4914	1.2346		0.6993
0.025	0.9091	2.1505		1.1561
0.015	1.1236	2.1978	3.1746	
0.015	1.0989	2.0000	2.5641	
0.0075	1.9417			
0.00375	3.4483			

Table 13: Baracarb fluid loss test on 11 micron filter paper

The figure below shows a comparison of the flow rate of 6% KCl brine through 25 micron filter paper by adding various grades and concentrations of Baracarb. By decreasing the Baracarb concentration the flow rate increased and therefore fluid loss control became worse.

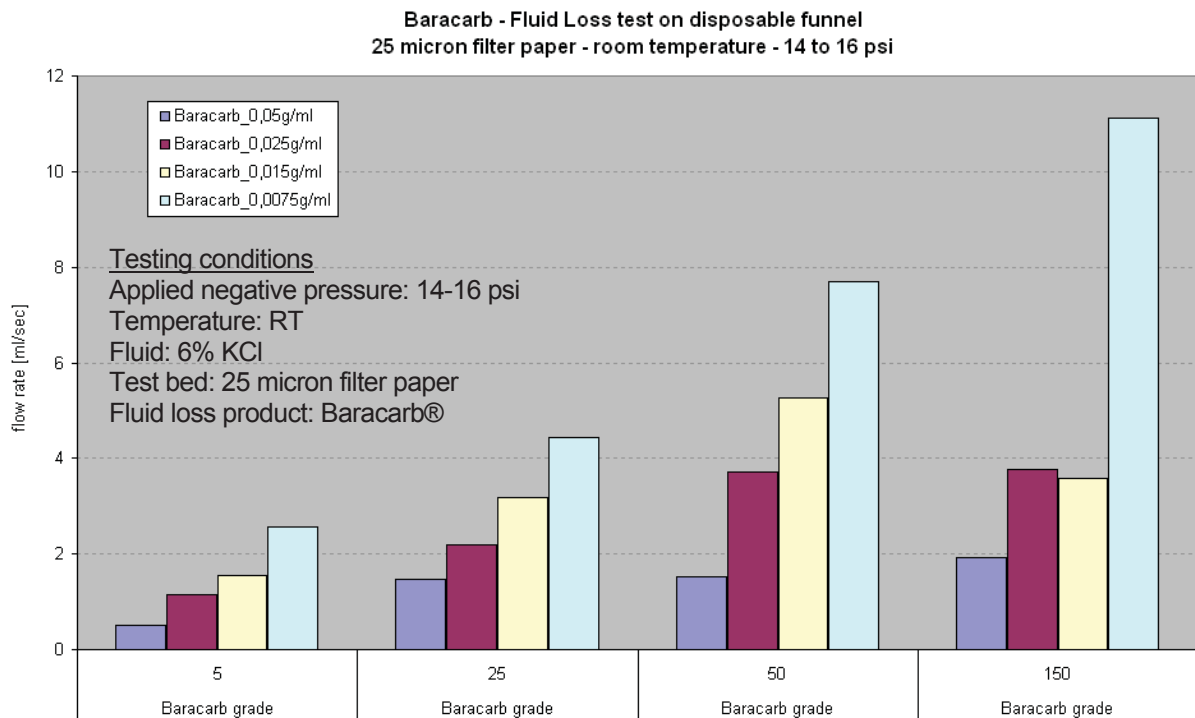


Figure 67: Baracarb fluid loss test on 25 micron filter paper

25µ filter paper	Baracarb grade 5	Baracarb grade 25	Baracarb grade 50	Baracarb grade 150
concentration [g/ml]	[ml/sec]	[ml/sec]	[ml/sec]	[ml/sec]
0.05	0.5013	1.4815	1.5152	1.9231
0.025	1.1429	2.1978	3.7037	3.7736
0.015	1.5385	3.1746	5.2632	3.5714
0.0075	2.5641	4.4444	7.6923	11.1111

Table 14: Baracarb fluid loss test on 25 micron filter paper

Below pictures show Baracarb® filter cakes. The filter cake on the left is a mixture of Baracarb 5 and Baracarb 25; 2.5 grams of each grade were used for this filter cake.

Right hand a filter cake made in the HPHT cell is pictured. As Baracarb 5 showed the best fluid loss control ability during the basic product evaluation it has been chosen for the HPHT fluid loss tests.



Figure 68: Baracarb filter cake on 11 micron Whatman filter paper



Figure 69: Baracarb filter cake on 20 micron ceramic filter disc

3.4.2 HPHT fluid loss tests

The following HPHT tests were conducted at room temperature and a pressure of 100 psi. Three different Baracarb concentrations have been tested in 400 ml of 3%KCl brine.

Due to the fast fluid throughput it was difficult to record a fluid loss curve over time. As already seen on the filter papers Baracarb reduced the flow rate significantly also through the 20 micron filter disc. From an average 23.5 ml/sec without fluid loss additives the flow rate was reduced to 6.6 ml/sec by using 30g of a mixture of Baracarb 5, 25 and 50.

As Baracarb 5 showed the best fluid loss control ability on the filter papers it was used for further HPHT tests at a concentration of 0.05 and 0.015 g/ml. Again a reduction in flow rate but no seal could be observed during this test.

The results of these tests are comparable to the results obtained by testing FDP-S875 at room temperature and giving the particles only 2 minutes to soak in brine. In this case also the FDP-S875 does not show a shut off fluid flow because temperature and time does not allow the product to crosslink.

3.4.3 Conclusion

All tested Baracarb grades provide good fluid loss control. The tests showed that fluid loss is slowed down significantly but no shut off could be seen. This may result from not meeting the exact recommendations of the average bridging particle size. As far as the performed test are concerned it can be concluded that Baracarb is not suitable for using it as a diverting agent based on the concept of high spurt loss and low C_w . Using this product the resin would not be transported into the formation as spurt loss but continuously at a reduced rate after the bridging agents had built up a filter cake on the formation wall. Further tests would be necessary if Baracarb creates filter cakes of equal permeability on various formation permeabilities. If it is able to equalize flow this way it can be used as diverting agent. Further as the Baracarb size distribution was not ideal throughout the tests the filter cake build up with recommended particle size should be tried to evaluate if total seal is possible and keeping spurt loss in mind also how quick it can be established.

The objective of these tests were to determine whether the concept of pumping resin and diverting agent in one stage and consolidate the formation with the resin spurt loss is possible. After testing the product behaviour of Baracarb on filter paper and in the HPHT cell it can be concluded that this concept does not favour the use of Baracarb. It is possible that Baracarb would favour the equalization of flow per unit thickness and lead to uniform resin distribution in a concept like this.

3.5 Testing of Polyacrylamide Gel as a Diverting Agent

The suitability of polyacrylamide gels as fluid loss control additives has already been tested at Halliburton. During these previous studies the influence of gel concentration, gel hardness and particle size on the gels fluid loss control ability has been analysed.

Within this work HPHT fluid loss tests were conducted with polyacrylamide gels of different rigidness. The objective is to determine the gels ability to generate a spurt loss so high to transport the resin far enough into the formation to reach a reasonable consolidation radius around the wellbore.

3.5.1 Preparation of the Polyacrylamide gel particles

The polyacrylamide gels were prepared by pouring 200ml water into a jar. By using a magnetic stirring device the water is stirred while adding all the other ingredients. Ten grams acrylamide are added to the water followed by bisacrylamide. The bisacrylamide is used as the cross linking agent

in this polymerization. By varying the amount of bisacrylamide the degree of cross linking and therefore the rigidness of the gel can be altered. While adding 0.05g bisacrylamide gives a soft gel which is very flexible and hard to break, adding 0.2g bisacrylamide creates a rigid gel which is easy to chop up. Adding potassium persulfate initiates the polymerization and addition of tetra ethyl ethylene diamine speeds up the reaction and allows polymerization at room temperature.

200 ml	Water	
10 g	Acrylamide	
0.05 g (soft gel) 0.1 g (medium gel) 0.2 g (rigid gel)	Bisacrylamide	Cross linker
0.1g	Potassium Persulfate 99+% A.C.S reagent K ₂ S ₂ O ₈	Initiator
5 drops	TEED (Tetra ethyl ethylene diamine)	Accelerator

Table 15: Polyacrylamide gel recipe

As soon as the gels are ready (Figure 70) water gel dispersions can be prepared. In order to get 10 Vol% gel dispersion 10g of polyacrylamide gel are added to 90ml of water. Gel and water are put into a plastic beaker and chopped up with a Silverson emulsifier (Figure 71). This results in 100ml gel-water dispersion with a polyacrylamide gel concentration of 0.1g/ml.

Samples of all three gel textures were prepared at polyacrylamide gel concentrations of 0.05g/ml, 0.1g/ml, 0.15g/ml, and 0.2g/ml. By diluting the dispersion with water the gel concentration in the system can be varied. It is going to be tested if the spurt loss can be adjusted by variation of gel concentration and if it is high enough for the purpose of resin diversion according to the concept described in chapter 3.1.



Figure 70: Polyacrylamide gel samples



Figure 71: Silverson

3.5.2 HPHT fluid loss tests

According to the Halliburton report „Crosslinked Polymer Hydrogels as Fluid loss Control Additives (II)“ from August 2006 static fluid loss tests with Polyacrylamide gels were performed on 10 micron sand discs (permeability about 0.95 D). Series of tests at room temperature as well as at 180°F were performed. The pressures used were 500 and 1000 psi. The volume fraction of gel added to the water was between 20 and 30%. The purpose of these previous tests was to analyse the suitability of these hydrogels for applications as frac packing and hydraulic fracturing. The

requirements for these applications include a low spurt loss and a low value for C_w . The best results in meeting these requirements were obtained with mixtures of rigid and soft gel. It has been concluded that the rigid particles do not deform and penetrate the sand disc but instead build a rather thick filter cake on the disc surface. The soft gel particles on the other hand may partially deform and penetrate the filter disc and therefore build a thinner filter cake and also reduce the pore spaces within the filter cake. The results for C_w found in the previous tests range between 0.0098 and 0.00294ft/sqrt(min). The lowest spurt loss was 0.02201gal/ft² and the highest 1.39008gal/ft², but this value seems to be an exception because most of the other spurt values are below 0.19806gal/ft².^[17]

As the gel particles have about the same density as water it is not necessary to use a stirring device for the HPHT tests. Other than that the procedure of the fluid loss tests remain the same as described in chapter 3.2.2. In order to be able to compare the results obtained from the PAm gel tests to the testing of FDP-S875 and Baracarb® the pressure applied during these tests is reduced to 300psi. As the suitability for the application of PAm gel particles within this particular diversion concept depends on the amount of spurt loss the concentration of PAm gel is reduced to a range of 3 – 6 Vol% PAm gel particles. First the behaviour of the different gel textures at the lowered concentrations was evaluated. As the mixture of hard and soft particles showed good results in terms of low spurt loss it was tried to evaluate which particle texture may result in high spurt values at lowered concentration. For part of the tests the gel particle size was estimated using a microscope. All tests were performed on 20 micron filter discs showing a permeability of about 2.8D.

Table 16 gives an overview on the gel concentration used and the resulting values for spurt and C_w . Compared to the tests of 2006 the reduction of gel particle concentration led to an increase of C_w which indicates worse fluid loss control over time. Generally speaking the spurt loss showed a slight increase compared to the previous testing but still remained very low. Due to the changes in gel particle concentration pressure and filter disc pore diameter it is not possible to relate these test series directly to each other.

PAm gel concentration [g/ml]	range of particle size [microns]	Spurt [gal/ft ²]	C_w [ft/sqrt(min)]	gel texture	Pressure [psi]	Temperature [°F]
0.066666667		0.04763593	0.0190901	rigid	300	Room temp (RT)
0.05	10-100	0.11804396	0.01688244	rigid	300	RT
0.038461538	10-100	no seal		rigid	300	RT
0.037037037	75-500	no seal		rigid	300	RT
0.033333333		no seal		rigid	300	RT
0.016666667		no seal		rigid	300	RT
0.05	25-100	0.2820855	0.00766207	medium	300	RT
0.05		0.19054372	0.00952611	medium	300	RT
0.048780488		0.09835073	0.00896077	medium	300	RT
0.040540541	25-500	0.63259033	0.02619991	medium	300	RT
0.020833333		no seal		medium	300	RT
0.05	10-50	0.8570346	0.09543328	soft	300	RT
0.045454545		0.32243642	0.00877691	soft	300	RT
0.04		no seal		soft	300	RT
0.04	25-100	no seal		soft	300	RT
0.0375	10-50	0.25189444	0.21819028	soft	300	RT
0.03125		no seal		soft	300	RT

Table 16: Overview of the results of testing with PAm gel particles on 20 micron discs

What the results regarding spurt loss mean for the concept of fluid diversion shows the following example. In a formation with 30% porosity the highest spurt value resulting from the tests (0.857gal/ft²) would be able to consolidate 0.25 ft near wellbore environment of a 6 inch wellbore. Compared to the high spurt values obtained from testing with the FDP-S875 at a temperature of 115°F the spurt losses achieved with the chopped PAm gel are much lower.

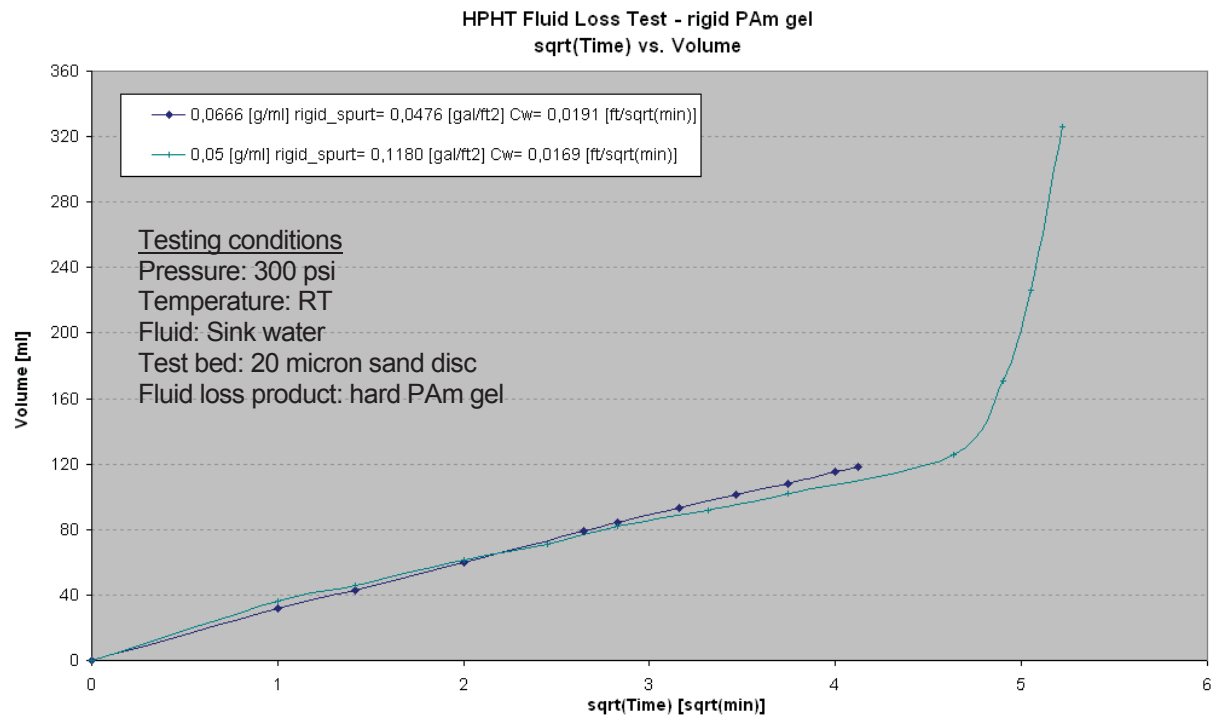


Figure 72: Fluid loss over sqrt(time) using rigid PAm gel particles

Detailed information on the testing conditions of the testing series is stated on the diagrams showing fluid loss over square root of time of the various PAm gel textures. Further it has to be mentioned that a possible fluid breakthrough during testing is not taken into account for calculation of spurt loss and C_w . This is not necessary because an actual leak in a downhole situation would be plugged by the particles continuously pumped past the leak during the duration of a job. So as an example in the figure above the last three data points are not included in the calculation of spurt and C_w value.

The first test within the testing series with Polyacrylamide gel was performed with the most rigid PAm gel particle at a concentration of 0.0667g/ml. Figure 72 shows the result of this test which gave a spurt value of 0.0476gal/ft² and a C_w of 0.0191ft/sqrt(min). This spurt value is far below the values that could be reached by using the FDP-S875. In order to get a higher spurt loss the concentration of PAm gel particles was further reduced. With a decrease in concentration to 0.05 g/ml the spurt loss increased by a factor 2.48 and also a slight decrease in C_w could be seen (Table 16 and Figure 72). Previous to this test the size range of the gel particles was determined by using a microscope. For this specific test the gel particle size ranged between 10 and 100 micron whereas the major part of the particles was between 10 and 25 micron. This particle size range results from chopping the Polyacrylamide gel at 4800rpm. The particles are shaped irregularly and the particle size will generally decrease with increasing the Silverson shear rate for chopping the gel.

The previous two tests using the gel containing the highest amount of cross linker (0.2g Bisacrylamide) showed spurt values lower than desired for this diversion concept. Therefore the PAm gel concentration was lowered for further testing. Fluid loss tests at lowered concentrations showed it is not possible to build up a sealing filter cake on the surface area of the sand disc by using a gel particle concentration of 0.037g/ml with gel particles ranging between 75 and 500 micron. Another test was performed at a slightly higher particle concentration (0.0385g/ml) but with smaller gel particles which showed a narrower particle size distribution than the previous sample. Unfortunately also the smaller gel particles (10-100 micron) did not seal off the filter disc surface area (Table 16). As no deformation of the rigid gel particles is expected the filter cake will be more porous with increasing gel particle size. In both samples (75-500 micron and 10-100 micron particle size) the gel particles were too large to create a sealing filter cake with the rigid gel particles.

Below pictures show the filter cake formed by the rigid Polyacrylamide gel particles. As described in the work done in 2006 the rigid gel particles form a thick filter cake. The particles are too stiff to deform and enter the disc pore space as well as too stiff to deform and create a less porous filter cake.



Figure 73: Filter cake made from the rigid Pam gel



Figure 74: Filter cake made from the rigid Pam gel – side view

The medium stiff PAm gel particles contain compared to the rigid gel only half the amount of cross linker. The tests performed can be looked up in Table 16 and the successful ones are plotted in the diagram below.

In general a decrease in C_w takes place when using the medium stiff gel particles compared to the rigid ones. The particles ability to deform to a certain extent into void spaces leads to a denser filter cake and therefore better fluid loss control over time. While for the rigid particles the C_w was slightly below $0.02\text{ft}/\sqrt{\text{min}}$ it drops below $0.0095\text{ft}/\sqrt{\text{min}}$ with the medium particles. In below diagram it can be seen that the violet graph, which shows the fluid loss record for the lowest gel particle concentration, has a steeper slope. For this particular test only half the surface area of the filter disc has been used. The intention behind sealing half the filter disc with epoxy resin was to reduce the flow rate through the filter disc and therefore succeed in establishing a sealing filter cake by using the same amount of particles and fluid volume (which as mentioned before is limited by the capacity of the HPHT cell). This method was supposed to give the particles, which have about the same density as water, more time to settle and build a filter cake before the entire fluid is lost. This lowest gel concentration ($0.04054\text{g}/\text{ml}$) resulted in the highest spurt loss ($0.633\text{g}/\text{ft}^2$) but also in the worst fluid loss control over square root of time. In this case C_w was with $0.0261\text{ft}/\sqrt{\text{min}}$ even slightly higher than for the rigid particles. The explanation for that might be the particle size distribution of this gel dispersion sample. Microscopic analysis showed that the major part of the particles was between 100 and 500 micron and only few particles were as small as 25 to 75 micron. This sample was prepared by chopping up the gel at 5800 rpm. In order to get smaller particles the rotations per minute (rpm) of the Silverson device has to be increased. While chopping up the medium gel at 5800 rpm produces particles within a size range of 25-500 micron it is possible to get a size distribution as narrow as 10-100 micron at 4800 rpm for the rigid gel samples. To obtain a particle size distribution from 25 to 100 micron the medium gel has to be chopped up at 6200 rpm.

Compared to the testing performed with the rigid particles the spurt values increased within testing the medium cross linked gel particles in every case but one. Therefore it can be stated that under the same testing conditions the medium gel particles showed better fluid loss control over time as well as for this concept favourable higher spurt loss.

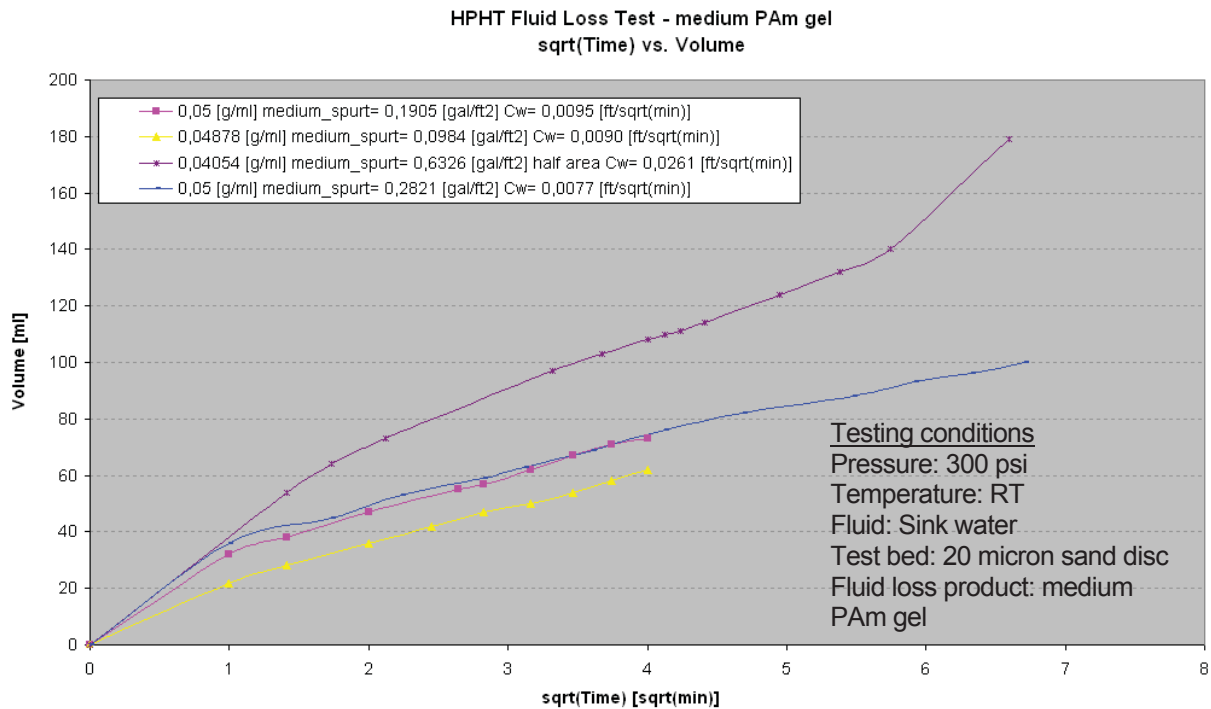


Figure 75: Fluid loss over sqrt(time) using medium PAm gel particles

In order to get an idea which gel texture is the most suitable for this diversion concept also the soft gel particles have been tested in the HPHT fluid loss cell.

In the diagram below the results of the tests performed with the least cross linked gel particles are plotted. This gel is really soft and flexible and therefore it is hard to separate samples from the jar. As stated in Table 16 two gel samples have been chopped up to a pretty narrow particle size distribution (10-50 micron). To obtain this size range the Silverson shear rate was adjusted to 7100 rpm. Using the rigid gel particle size ranges of 10-100 micron and 75-500 micron at a concentration of about 0.037g/ml did not seal off the 20 micron filter disc. As stated before too large gel particles and their rigidity could be the explanation. Therefore it has been decided to use a concentration of 0.0375g/ml but smaller gel particles (10-50 micron) for the first test with the soft gel. As the brown graph in Figure 76 indicates the established fluid loss control over time was not good ($C_w = 0.218\text{ft}/\sqrt{\text{min}}$) and due to the steep slope of the graph also the spurt loss value is with $0.252\text{gal}/\text{ft}^2$ on the low side. For this particular test again only half the surface area of the filter disc has been used. Again the intention is to delay fluid loss rate by reducing the filter area and therefore allow the filter cake to build up before the fluid volume is lost. As below graph shows most of the water in the HPHT cell has been lost within about four minutes. So compared to the same concentration of rigid particles the reduction in size and surface area at least slowed down the flow rate and it could be managed to record some data points. In order to figure out if the same particle size distribution (10-50 micron) would establish a better seal the concentration for the next test has been increased to 0.05g/ml. In Figure 76 the result is represented by the pink graph. Compared to the lower concentration the C_w value decreased to $0.095\text{ft}/\sqrt{\text{min}}$ and therefore the trendline connecting the data points recorded after the filter cake build up leads to a higher intersection on the ordinate. So the spurt loss value increased to $0.857\text{gal}/\text{ft}^2$. Although one test was performed using half the sand disc area as filter medium the fluid volume has been lost in both tests within about four minutes. It was not possible to establish a satisfying seal using soft particles of a size between 10 and 50 microns on the 20 micron disc. The turquoise coloured graph (representing a gel particle concentration of 0.0454g/ml) in below diagram showed the best results regarding fluid loss control over square root of time. For this sample the gel was chopped up at a shear rate of 6200 rpm, which means that the particle size is above those of the previous soft gel samples. No microscopic analysis of the gel particles has been done.

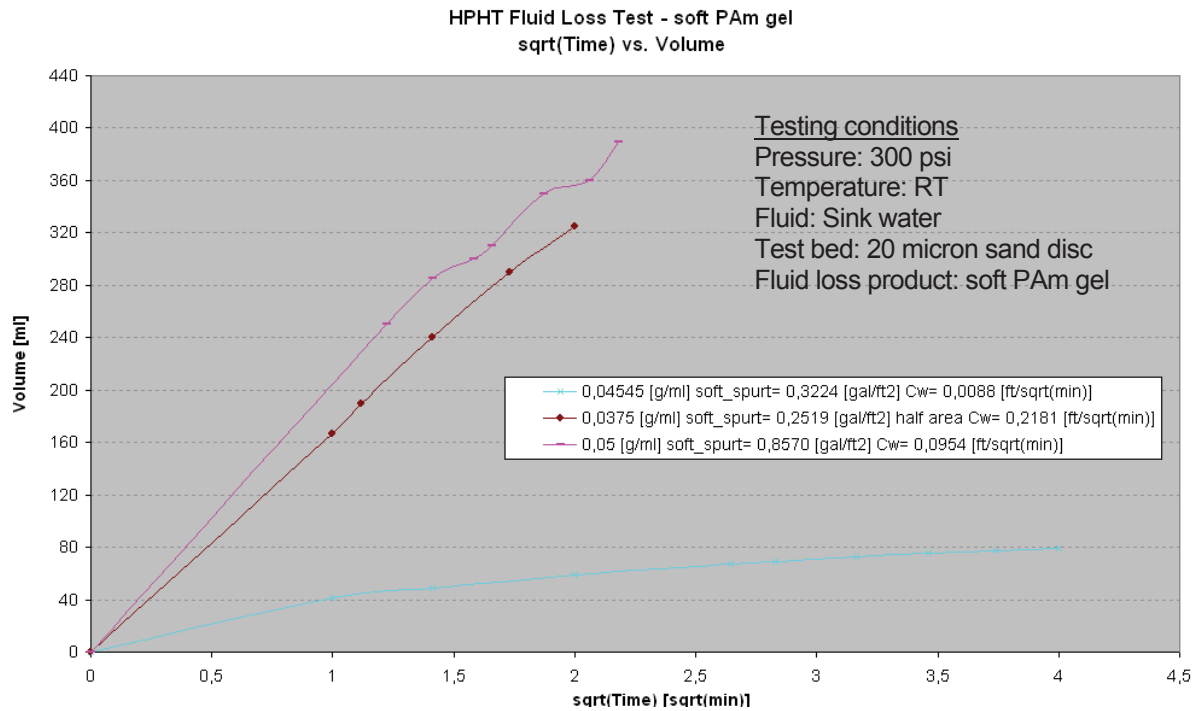


Figure 76: Fluid loss over sqrt(time) using soft PAm gel particles

3.5.3 Conclusion

Testing of three different Polyacrylamide gel textures chopped up to various particle size ranges were tested based on the previously defined diversion concept. In 2006 adding 20-30 Vol % of PAm gel and the mixture of soft and rigid gel particles was defined as suitable to obtain good fluid loss control and low spurt loss. Therefore the tests performed in the course of this work focused on the various gel textures and their fluid loss behaviour at lower particle concentration. It can be summarized that spurt losses did not exceed 0.875gal/ft² and are more likely to be in the range of 0.1 to 0.3gal/ft². According to Table 4 a spurt loss of 1.7gal/ft² is necessary to obtain a resin penetration depth of 0.39ft in a 5in wellbore and a reservoir of 30% porosity. This example shows that the spurt loss obtained during this testing series is too low to for the defined diversion application.

As defined in chapter 3.1.1 a C_w value of 0.005ft/sqrt(min) would be an acceptable value for fluid loss control over square root of time. Without exception all C_w values exceed this benchmark. The lowest result for C_w obtained is 0.0077ft/sqrt(min). The flow rate through the 20 micron filter disc has been reduced throughout the tests but no total seal was achieved. To obtain total seal more precise particle size analysis has to be performed and it is necessary to produce samples showing a wide size distribution including particles as small as necessary to plug void spaces in the filter cake. This might be more necessary for the rigid gel samples because the soft PAm gel is able to deform and therefore plug pores.

3.6 Impact of Permeability

As concluded at the end of the description of the HPHT fluid loss tests performed with FDP-S875, Baracarb® and the Polyacrylamide gel only FDP-S875 is able to transport a treatment fluid into a formation via spurt loss. Baracarb® and the Polyacrylamide gel particles achieved very small spurt loss values compared to those of FDP-S875. As it is the most promising candidate to be tested for its compatibility with a water based resin further tests will continue only with FDP-S875.

First of all there has been another prerequisite defined within the diversion concept: The spurt loss obtained with the fluid loss additive should not depend on the permeability of the filter medium. The

ideal scenario would be total independence of permeability and the only factor defining spurt loss would be the fluid loss additive concentration. As previous testing has shown it is difficult to directly link the product concentration to the resulting spurt loss. Within some testing series it has been possible to see certain trends but exact predictions are not possible.

What could be found was an amount of FDP-S875 per unit area that is able to seal of the 20 micron filter cake (0.193g/in² or 0.0299g/cm²). This filter disc has a permeability of 2800mD or 2.8D. Within this chapter it is described whether the same amount of product will seal off also discs of higher and lower permeability and how the change in permeability affects the spurt loss values obtained and the wall building coefficient C_w.

Below table gives an overview on the four permeabilities available for testing. This permeability data has been provided by Baroid another Halliburton supplier. Of course these values are averages and every sand disc would show individual permeability values, but this data is sufficient to get an idea of the available ranges and the permeability difference between the sand discs of various pore diameters.

average disc pore diameter [micron]	permeability [mD]
5	750
10	950
20	2800
35	5500

Table 17: Permeabilities of available filter discs

Testing was continued at 300psi and the temperature chosen for the testing series on various permeable sand discs was 115°F. As the best results have been achieved with a FDP-S875 soaking time of 2 minutes this procedure was used further on. To be on the safe side and in order to have a representative amount of comparable data sets on the 20 micron disc the following test were performed with a particle concentration of 0.001875g/ml. The fluid system remained 6% KCl brine.

So far only 20 micron filter discs have been used for fluid loss tests. Below images show filter cakes created by using the mentioned S875 concentrations on a 5, 10 and 35 micron sand disc. The 5 micron disc is shown in Figure 77; it is white and its surface is visibly finer than those of the 20 and 35 micron disc. The 10 micron disc can be recognized by its light grey color and with a permeability of 950mD this disc is has a slightly higher permeability than the 5 micron disc with 750mD. A significantly higher permeability shows the 35 micron Fann filter disc. With its permeability of 5.5D it is about twice as high as the permeability of the 20 micron disc and about 5.8 times higher than those of the 10 micron disc. And for comparison the permeability of the 5 micron disc would have to increase by a factor 7.3 to reach the value of the 35 micron disc. Despite these differences the filter cakes on the 5, 10 and 35 micron discs look similar to those created on the 20 micron disc under the same testing conditions.



Figure 77: 0.75g FDP-S875 on a 5 micron filter disc



Figure 78: 0.75g FDP-S875 on a 10 micron filter disc



Figure 79: 0.75g FDP-S875 on a 35 micron filter disc



Figure 80: 0.75g FDP-S875 on a 35 micron filter disc – top view

If their fluid loss control quality over time is as good as on the 20 micron disc and to which extent the permeability changes influence the spurt values obtained is described in the following.

3.6.1 Impact of permeability on spurt loss

The following diagrams display the fluid loss behaviour over time on 5, 10, 20 and 35 micron filter discs. Below the four diagrams showing the fluid loss curves a table summarizes all results obtained from these tests.

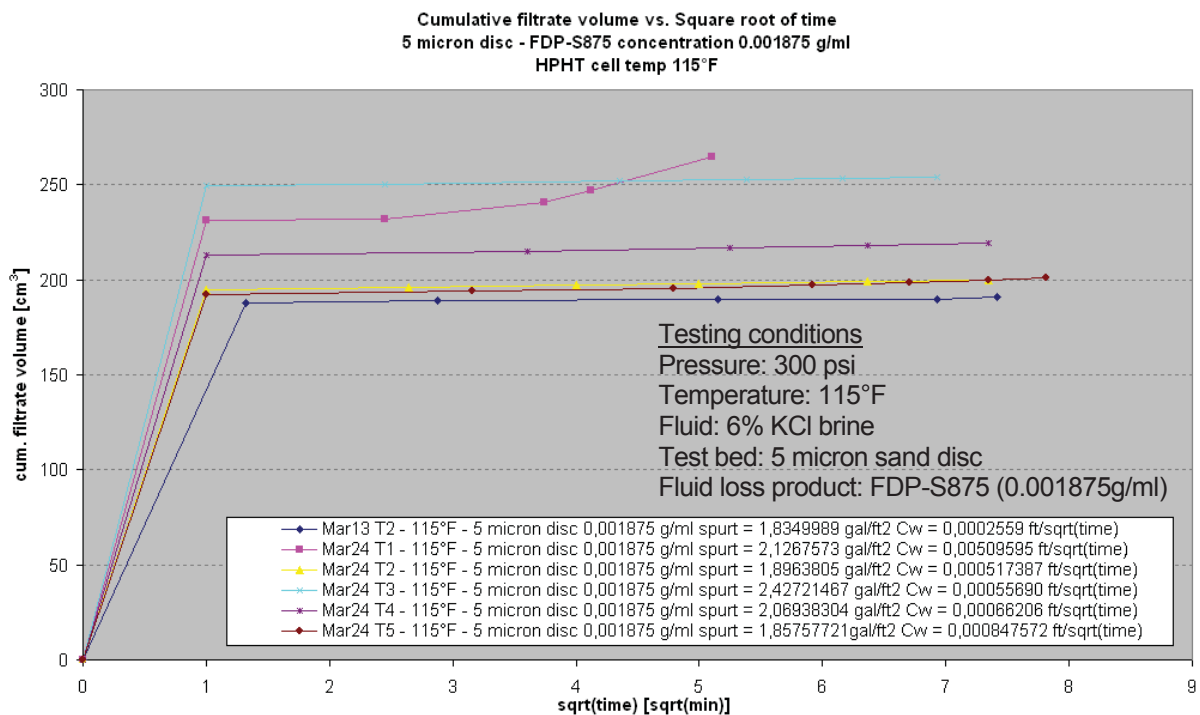


Figure 81: Fluid loss behaviour over sqrt(time) on 5 micron filter discs

Figure 81 plots the results of the HPHT tests on the 5 micron discs. Six tests were performed under the conditions described in chapter 3.6. With the exception of the pink graph (test Mar24_T1) all tests showed very good sealing ability over about 50 minutes. In the case of the pink graph a fluid breakthrough occurred and therefore increased the C_w value which is calculated by using the slope m of the graph. All other values of C_w are very good, because all of them are below 0.00084

ft/sqrt(min). Three tests seem to create a trend in cumulative fluid loss and spurt (Mar13_T2, Mar24_T2 and Mar24_T5); the spurt value of these tests lie around 1.85gal/ft². The highest spurt value was obtained in test Mar24_T3 and is as high as 2.43gal/ft².

Figure 82 shows the results of the tests on 10 micron filter discs. By comparing it to the previous figure it can be seen that the yellow graph (spurt loss = 2.47gal/ft²), which shows the least cumulative fluid loss, corresponds to the highest cumulative fluid loss in the testing series on the 5 micron discs (Mar24_T3, spurt loss = 2.43gal/ft²). The other fluid losses and spurt values on the 10 micron discs are higher than those on the smaller ones. Again a trend has been created. Three out of five tests gave spurt values around 2.74gal/ft².

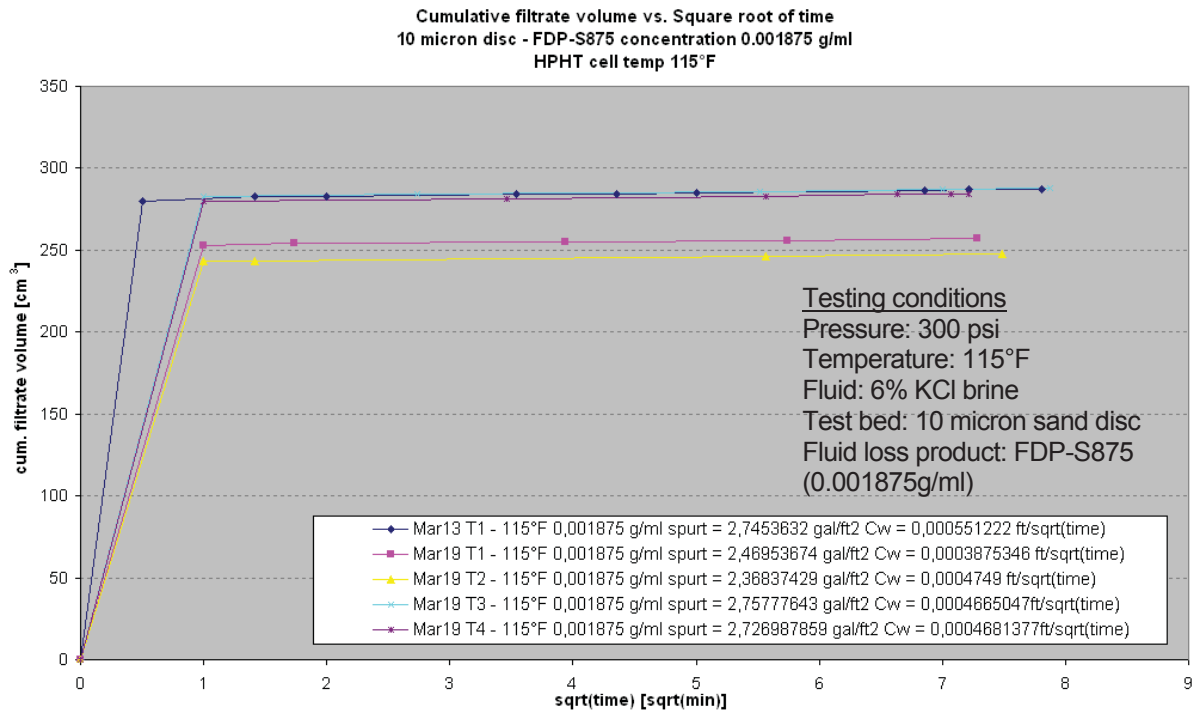


Figure 82: Fluid loss behaviour over sqrt(time) on 10 micron filter discs

In the following figure all tests performed on 20 micron filter discs are plotted. It can be seen that within these nine tests no trend could be created which could give estimation in spurt loss obtained by following tests. Twice it was possible to reproduce results obtained before. Mar12_T6 and Mar12_T7 gave the same results in cumulative fluid loss and both spurt values are about 2.86gal/ft². Mar17_T3 was able to reproduce the results of Mar17_T2. Both tests resulted in the second highest cumulative fluid loss over time and also the second highest spurt loss (about 3.46gal/ft²). The highest spurt loss was obtained with test Mar11_T1 and is as high as 3.66gal/ft². During this test 375ml brine has been lost within the first 45 seconds after opening of the discharge valve. Within the remaining 36 minutes only 3.6 more milliliters have been lost. This means after the spurt loss only 1/16 of the total 400ml 6% KCl brine remained in the cell body. This was the very best and highest spurt value obtained throughout all tests. Even at the 35 micron filter discs no higher spurt loss and cumulative fluid loss has been seen.

Figure 85 shows the fluid loss record of the testing on 35 micron discs. The cumulative fluid loss lies between 300 and 350ml which means it is below the five best results of the tests on 20 micron filter discs. The highest spurt value is 3.34gal/ft² compared to the 3.66gal/ft² from a previous test.

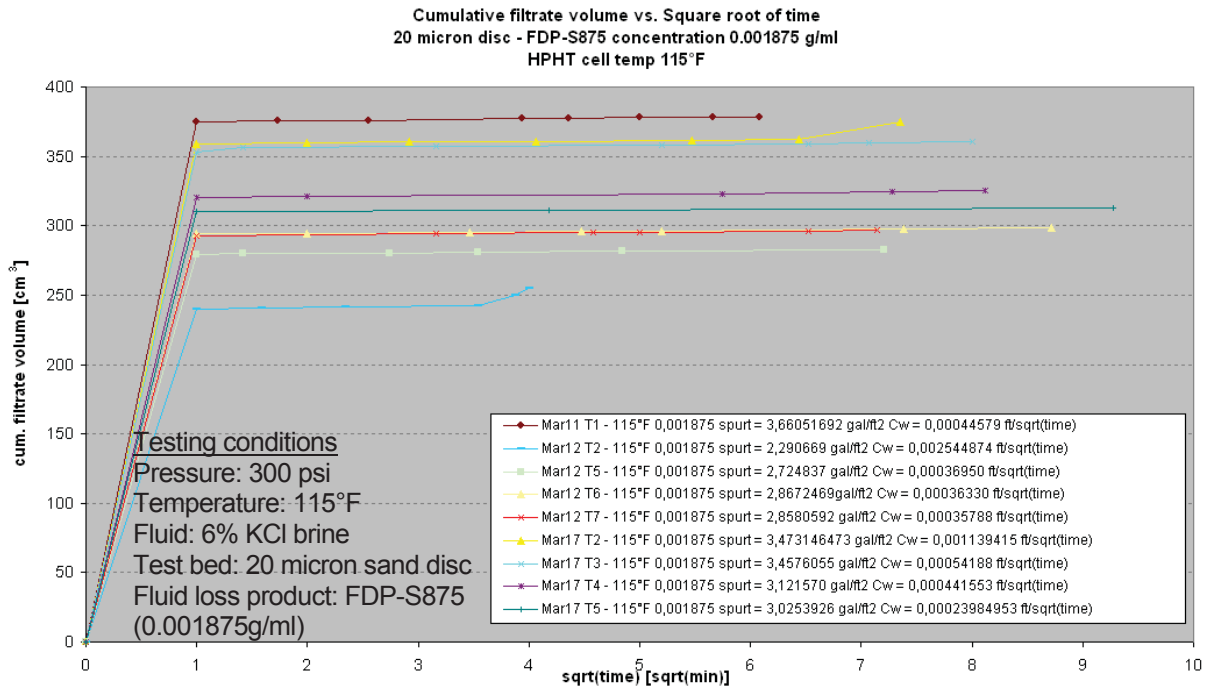


Figure 83: Fluid loss behaviour over sqrt(time) on 20 micron filter discs

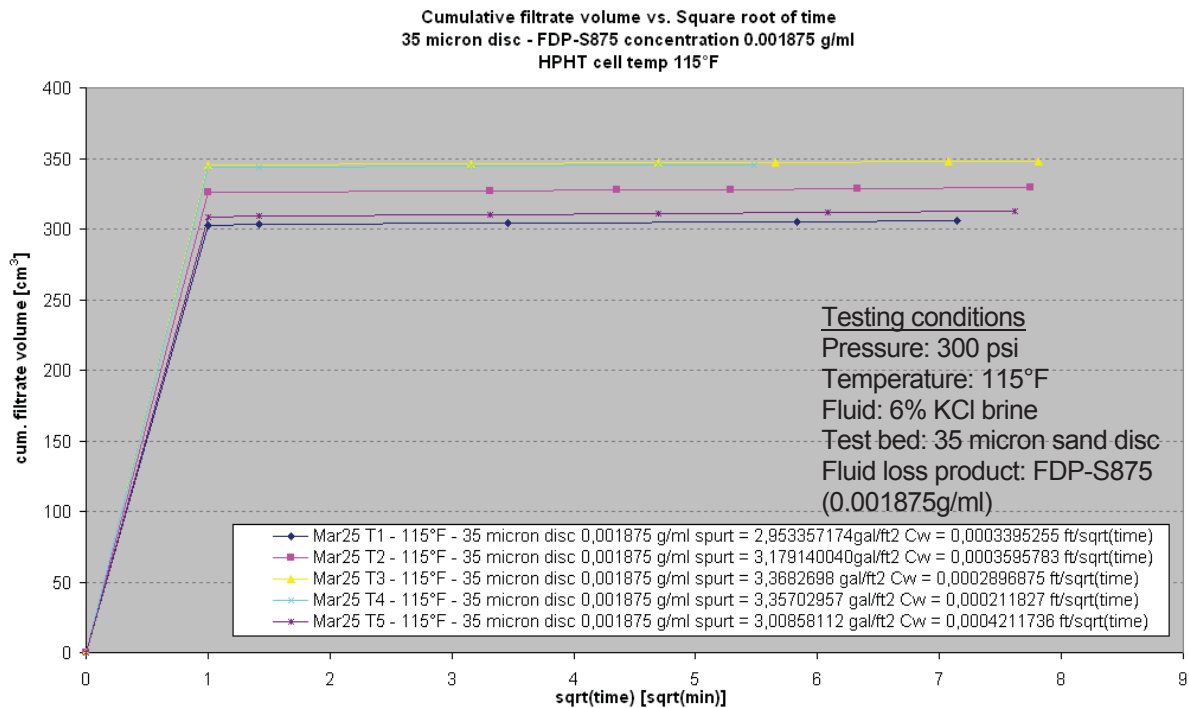


Figure 84: Fluid loss behaviour over sqrt(time) on 35 micron filter discs

In the following table all results are summarized. It starts with the filter discs having the highest permeability (35 micron) and goes down to the 5 micron disc. Within each disc size the results are arranged by decreasing spurt loss. The spurt value written in red is the highest obtained throughout testing; the ones marked by a red rectangle indicate trends created on certain disc permeabilities.

Test	average pore diameter of disc [micron]	temperature [°F]	pressure [psi]	concentration [g/ml]	spurt loss [gal/ft ²]	C _w [ft/sqrt(time)]
Mar25 T3	35	115	300	0.001875	3.3682698	0.000289688
Mar25 T4	35	115	300	0.001875	3.35702957	0.000211827
Mar25 T2	35	115	300	0.001875	3.17914004	0.000359578
Mar25 T5	35	115	300	0.001875	3.00858112	0.000421174
Mar25 T1	35	115	300	0.001875	2.953357174	0.000339526
Mar11 T1	20	115	300	0.001875	3.66051692	0.00044579
Mar17 T2	20	115	300	0.001875	3.473146473	0.001139415
Mar17 T3	20	115	300	0.001875	3.4576055	0.00054188
Mar17 T4	20	115	300	0.001875	3.12157	0.000441553
Mar17 T5	20	115	300	0.001875	3.0253926	0.00023985
Mar12 T6	20	115	300	0.001875	2.8672469	0.0003633
Mar12 T7	20	115	300	0.001875	2.8580592	0.00035788
Mar12 T5	20	115	300	0.001875	2.724837	0.0003695
Mar12 T2	20	115	300	0.001875	2.290669	0.002544874
Mar19 T3	10	115	300	0.001875	2.75777643	0.000466505
Mar13 T1	10	115	300	0.001875	2.7453632	0.000551222
Mar19 T4	10	115	300	0.001875	2.726987859	0.000468138
Mar19 T1	10	115	300	0.001875	2.46953674	0.000387535
Mar19 T2	10	115	300	0.001875	2.36837429	0.0004749
Mar24 T3	5	115	300	0.001875	2.42721467	0.0005569
Mar24 T1	5	115	300	0.001875	2.1267573	0.00509595
Mar24 T4	5	115	300	0.001875	2.06938304	0.00066206
Mar24 T2	5	115	300	0.001875	1.8963805	0.000517387
Mar24 T5	5	115	300	0.001875	1.85757721	0.000847572
Mar13 T2	5	115	300	0.001875	1.8349989	0.0002559

Table 18: Summary of all tests on various filter discs- 115°F and 0.001875g/ml

In Table 19 the x-fold increase in filter disc permeability when switching from one cake to another is shown. The permeability of a 10 micron filter disc is 1.27 times higher than those of a 5 micron disc. To relate the 5 micron permeability to those of a 20 micron disc it has to be multiplied by a factor 3.73 and to reach the permeability of a 35 micron disc it has to be a factor 7.33. Further values can be seen in the table.

x- fold increase in permeability				
average pore diameter [micron]	5	10	20	35
5	1	1,27	3,73	7,33
10	0,79	1	2,95	5,79
20	0,27	0,34	1	1,96
35	0,14	0,17	0,51	1

Table 19: x-fold increase in filter cake permeability

Keeping these permeability relations in mind the next figure and table become interesting. Figure 85 plots the calculated spurt values in gal/ft² vs. the average pore diameter of the filter discs (which is related to their permeability by Table 17). The first impression by looking at the diagram is that the difference in spurt loss values between the 5 and 35 micron disc is not as big as it could be expected by a 7.33 times higher permeability of the 35 micron disc. In Table 20 the differences in spurt loss values are summarized. To obtain the table values it has been calculated how many times the spurt values at 35 micron discs are higher than those of the 10 micron discs. The resulting

five values were used to calculate the arithmetic mean value which is in case of 10 and 35 micron discs 1.22. This means the spurt loss at a 35 micron filter disc is 1.22 times higher than at a 10 micron disc. Remembering that the permeability of the 35 micron disc is 5.79 times higher than those of the 10 micron disc this result is very satisfying. The permeability increase of a factor 7.33 between the 5 and 35 micron discs just lead to an average increase in spurt loss of a factor 1.54. What surprises is that despite the two fold increase in permeability from 20 to 35 micron discs the highest spurt values obtained from testing on the 20 micron disc are above those of the 35 micron disc. For calculating the average used in below table only the five highest spurt values of the 20 micron disc have been used. Therefore the table shows a 0.95 fold increase in spurt loss from 20 to 35 micron filter discs. By using the results of the tests number 5, 6, 7, 8 and 9 from below diagram to compare it to results on the 35 micron disc it would lead to a 1.16 fold increase in spurt loss. Considering the comparison of 20 and 35 micron discs shows that the spurt loss obtained with FDP-S875 does not necessarily depend on filter medium permeability.

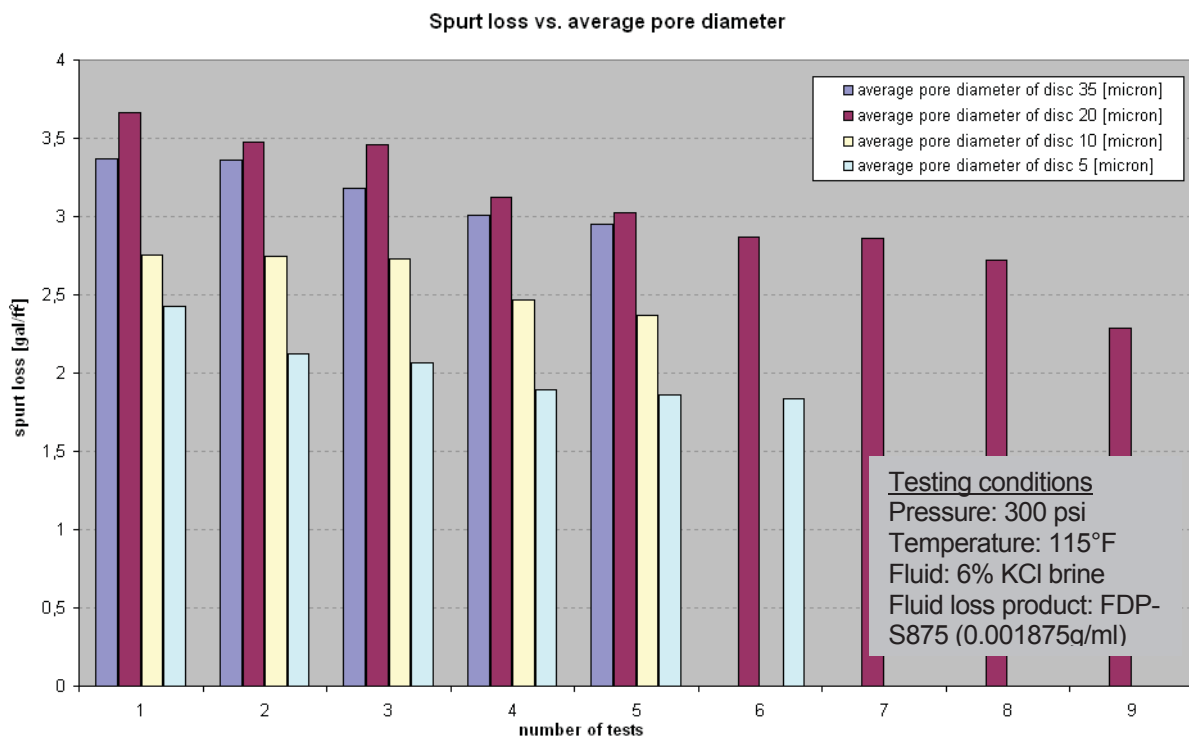


Figure 85: Spurt loss vs. average pore diameter of Fann filter discs

x-fold increase in spurt loss				
average pore diameter [micron]	5	10	20	35
5	1	1,26	1,62	1,54
10	0,79	1	1,28	1,22
20	0,62	0,78	1	0,94923721
35	0,65	0,82	1,05	1

Table 20: Averaged x-fold increase in spurt loss values (arithmetic mean)

The analysis of spurt loss on various filter disc permeabilities showed promising results. As mentioned before the ideal case would have been an independence of spurt loss value from filter disc permeability. This total independence could not be seen, but especially between 5 and 35 as well as between 10 and 35 micron discs it is much lower than it might have been expected. Comparing 5 and 10 micron discs it can be stated that the 1.27-fold increase in permeability resembles the average increase in spurt loss comparing these two filter discs (1.26-fold increase).

3.6.2 Impact of permeability on C_w

In this chapter the influence of variation in permeability on the wall-building fluid loss coefficient C_w is analysed. In the diagram below the wall building fluid loss coefficients obtained during the HPHT testing on various filter discs are plotted. It can be seen that the majority of C_w values are below 0.0006ft/sqrt(min). Exact values for all tests can be found in Table 18.

In general it can be concluded that the fluid loss control ability of FDP-S875 does not become worse with increasing or decreasing filter disc permeability.

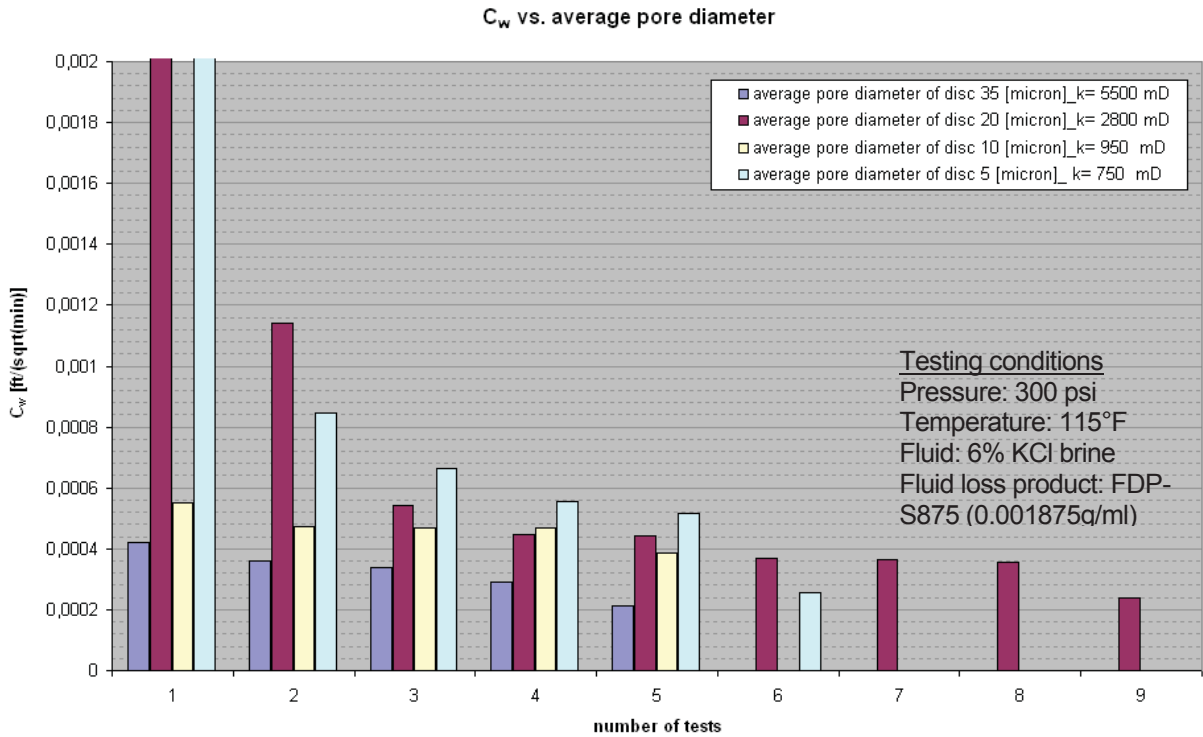


Figure 86: Influence of permeability on the wall building fluid loss coefficient C_w

4 Measurements with water based resin system

In chapter three the basic suitability of FDP-S875 for the defined diversion concept has been proven in KCl brine. Within this chapter testing is performed with a water based resin system.

The water based resin system used is composed by FDP-S867A-07 and FDP-S867B-07. According to its material safety data sheet (MSDS) FDP-S867A-07 consists of 10-30% epoxy resin and 1-5% potassium chloride. S867A is applied as the resin while S867B is applied as the hardener its ingredients are 1-5% potassium chloride and 5-10% polyamine polymer. The resin system is made by mixing equal amounts of part A and part B. FDP-S867A is put in a jar placed on a magnetic stirring plate, the fluid is stirred while adding an equal amount of hardener (FDP-S867B) to the resin.

This chapter includes the basic compatibility tests of FDP-S875 with the resin system, HPHT fluid loss tests on sand discs, HPHT fluid loss tests on unconsolidated sand packs, regain permeability tests and compressive strength tests on consolidated sand packs.

4.1 Compatibility tests

Providing sand control by chemical consolidation requires a product which is able to consolidate an unconsolidated formation to certain strength. Further it requires a diversion product which is able to distribute the resin uniformly over the entire productive zone. If for any reason a service company can not provide both they will not get the job.

Within this chapter compatibility tests between S875 and the resin system are described making sure that the fluid loss control ability of S875 is not impaired by the resin system. Further it has to be tested if the resin system itself acts as fluid loss additive and if the consolidation ability of FDP-S867A and B is somehow impaired by the fluid loss additive.

4.1.1 Impairment of fluid loss ability

For these tests the procedure and equipment described in chapter 3.2.3 was used. In resin at room temperature FDP-S875 needs about the same time to hydrate as before in the KCl solution. Both wet and dehydrated S875 products should be tested on filter papers.

As a first trial the flow of plain resin through the filter paper was tested. Impairment of flow could not be seen neither for resin at room temperature nor for resin heated up to 140°F. One specification of the resin is that as long as it stays below 175°F it will not separate from the solvent, precipitate and impair filtration through a porous medium.

The next step was to create FDP-S875 filter cakes in the resin system. 1.5 grams of dry FDP-S875 were put in 100ml resin at 136°F. The amount of resin left after finishing the fluid loss test was 58ml. This means the total fluid loss has been 42ml. The resin left in the disposable funnel was poured off and replaced by a little bit of brine in order to keep the filter cake hydrated while being in the oven. Then the sample was put in the oven and cured over night at 150°F. In an actual downhole situation it is necessary to remove the filter cake after the job is finished. As mentioned before FDP-S875 breaks by lowering the pH value. This means in order to break the created filter cake 100ml of 15% HCl preheated to 140°F were put in the funnel and vacuum pressure was applied. After soaking for 27min the acid was reheated and put on the cake again. Within this 27minutes 10ml of the HCl has been lost already. After another 1h and 50min the acid was reheated again and further fluid loss could be observed.

Another filter cake has been created by using the wet version of FDP-S875. Six grams wet S875 were added to 100ml resin heated to 130°F. The amount of resin left after finishing the fluid loss test was similar to the previous test (54ml). Again the resin leftover had been poured off and was

replaced with a little amount of brine. Then the sample was put in the oven and cured over night at 150°F. In order to destroy the filter cake and regain permeability through the filter paper 100ml of 15%HCl had been preheated to 130°F and was put on top of the filter cake. It could be observed that the 100ml HCl leaked through the filter cake in 3 hours. The HCl was reheated once after one hour. Again about 14-16psi pressure has been applied by the house vacuum line while the cake was treated with the acid.



Figure 87: wet FDP-S875/resin filter cake after HCl treatment



Figure 88: wet FDP-S875/resin filter cake after HCl treatment

Above Figures show two filter cakes after acid treatment. The HCl treated filter cakes of dry and wet FDP-S875 look similar as far as the top view is concerned. The internal filter cake textures of show differences. Figure 88 shows the fine internal texture of the filter cake created by the dry product. There is also a borderline visible which seems to separate the resin/FDP-S875 filter cake from a thin resin layer on the top of the filter cake. The internal texture of the cake created with the wet product is coarser than that of the dry product. In this case no separation between resin and fluid loss product can be seen with the naked eye.

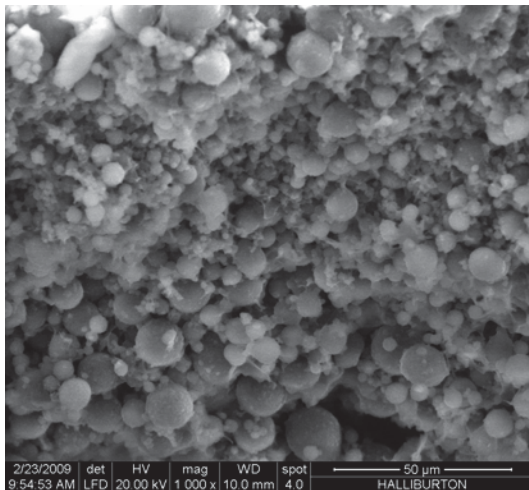


Figure 89: Sample surface area

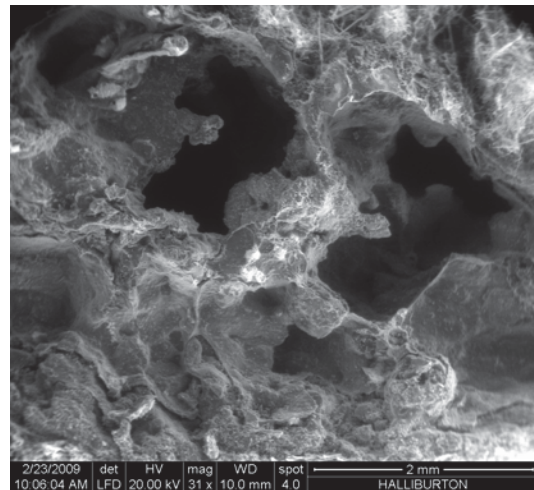


Figure 90: Bottom of the sample near filter paper

Filter cake samples have been handed to the analytical department for analysis. The following data and Figures 89 and 90 are created by the analytical department. In case of an untreated sample the center of the filter cake consist of a foamed material and the sample has an outer shell of small resin beads (1-25micron). Also some larger, irregular grains (size about 1mm) and some larger resin beads near the filter paper were detected. Figure 89 shows the spherical particles on top of the sample at 1000-fold magnification. Figure 90 gives an impression of the sample structure 4mm above the filter paper. The total height of the sample is 14mm (50-fold magnification). The acid treated sample has a structure similar to that of the untreated sample. The filter cake thickness is

16mm and again it showed a central core of foam and an outer coating which is dominated by small resin beads.

From the tests performed it can be concluded that the used resin system does not impair the fluid loss control ability of FDP-S875. The plain resin system does not act a fluid loss additive as long as kept below a certain temperature range. And it was possible to regain permeability of the filter cake after fluid flow had been shut off completely.

4.1.2 Impairment of consolidation ability

In order to exclude an interaction between resin and FDP-S875 leading to a failure of sand consolidation a very basic test has been performed. An unconsolidated sand pack of Oklahoma #1 sand in a glass pipe has been tried to be consolidated. In below figure the Oklahoma #1 sand pack is the white column, below 10g of filter sand (brown) should prevent that the Oklahoma #1 is sucked through the screen on bottom. The mixture of resin and S875 was poured on top of the brine saturated sand pack and vacuum pressure was applied. Part of the resin filtrated into the sand pack before the fluid loss additive created a filter cake and shut off filtration. The sand pack has been put in a 150°F water bath for several days in order to allow the resin to cure.

After curing the resin a regain permeability test has been done by pouring 150°F hot 15% HCl acid on top of the filter cake. Vacuum pressure was applied and by looking at the surface of the filter cake air bubbles could be observed, a sign that the acid is about to degenerate the guar filter cake. As seen during the testing on the filter papers also here it was possible to regain permeability of the sand pack.



Figure 91: Testing of consolidation ability of resin/FDP-S875 mixture

4.2 Fluid loss tests in HPHT cell

The previous testing showed the compatibility of FDP-S875 and the water based resin system using small resin volumes in very basic tests. In the following tests S875 is used in the HPHT cell at the same concentration as in the 6% KCl brine. Using the brine as fluid system favourable results were obtained at 115°F, 300psi using 0.75g S875 in 400ml (0.001875g/ml) and giving the fluid loss

additive a soaking time of about 2 minutes. The same concentration, temperature, pressure and testing procedure is used for the subsequent tests with the resin system.

Chapter 3.6.1 describes the dependence of spurt loss on various sand disc permeabilities using 6% KCl brine. Subsequently it is determined if the influence of different permeabilites is similar by performing the fluid loss tests with resin instead of brine. Below figure shows a plot of cumulative filtrate volume vs. square root of time of various filter disc pore sizes.

Both Figure 92 and 93 show clearly the reduction of spurt loss compared to the tests done with 6% KCl brine as fluid system.

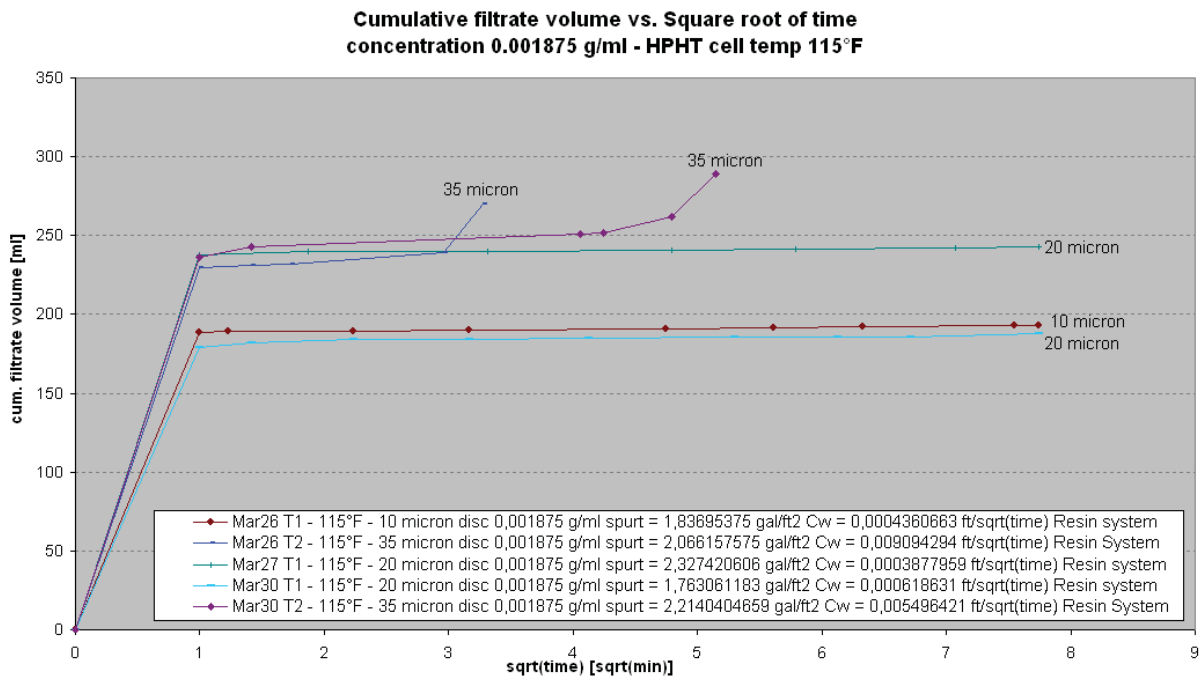


Figure 92: HPHT fluid loss test using water based resin as carrier fluid

The diagram below shows the spurt values achieved on four different sand disc sizes using 6% KCl brine and a S875 concentration of 0.001875 g/ml. In below plot only the two highest spurt values obtained during the test with KCl brine are plotted. Comparing those to the tests done with the resin system it can be seen that the spurt loss clearly decreases when the resin system is used as carrier fluid. The spurt values obtained on the 35 micron disc with the resin system are below those reached at the 5 micron disc using the brine as carrier fluid. This observation could lead to the assumption that the resin itself acts as a fluid loss control agent. In order to prove the conclusions obtained from the basic compatibility tests a fluid loss test in the HPHT cell was performed without adding S875. At 300psi and 115°F there is no impairment of flow created by the resin system. All tests showed clearly smaller spurt values for the resin system. The permeability of the 35 micron disc (5500mD) is approximately twice as high as those of the 20 micron disc (2800mD). The spurt loss on the other hand does not necessarily decrease in the tests performed on the 20 micron disc. The permeability of the 10 micron filter disc (950mD) is about 3 times lower than the permeability of the 20 micron disc. As it can be seen in the diagram and table below the spurt loss obtained from the test on the 10 micron disc is 1.837gal/ft². Comparing this value to those from the tests on the 20 micron discs (2.327 respectively 1.763gal/ft²) shows that the spurt loss does not strictly depend on the permeability of the filter medium.

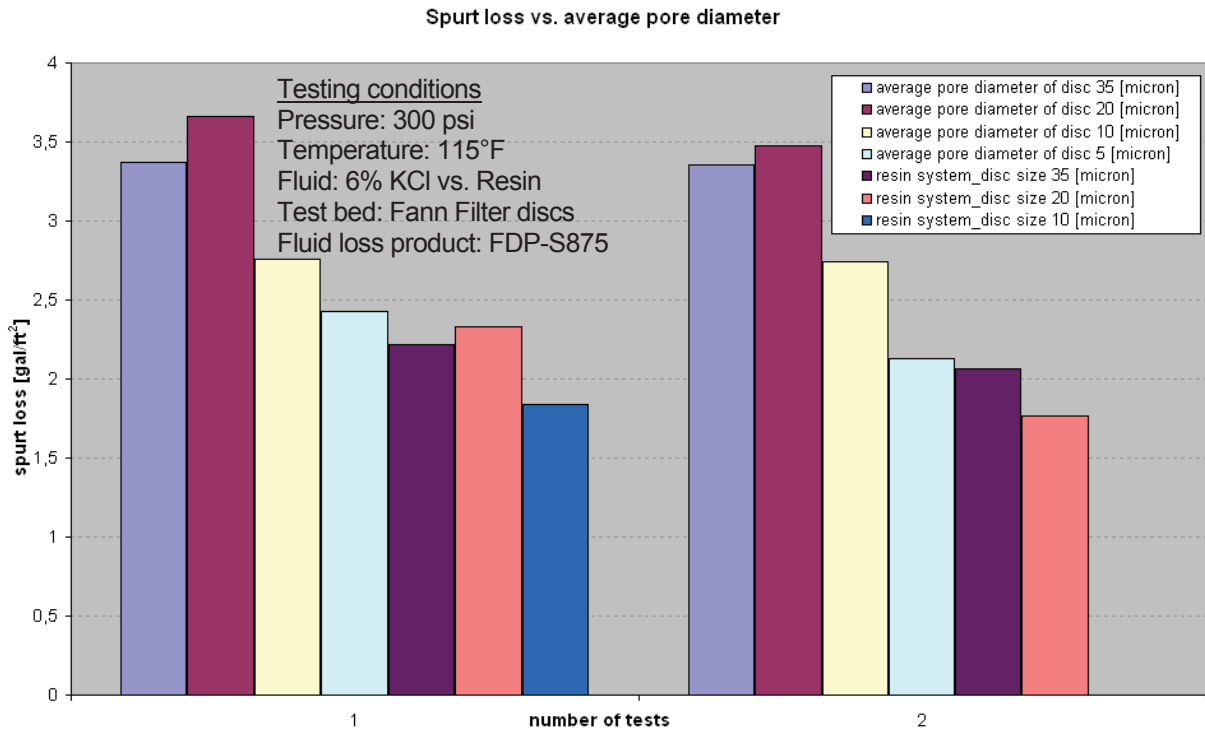


Figure 93: Comparison of spurt loss by variation of carrier fluid

	average pore diameter of disc [micron]	temperature [°F]	concentration [g/ml]	spurt loss [gal/ft²]	C _w [ft/sqrt(time)]
brine	35	115	0.001875	3.3682698	0.00028969
brine	35	115	0.001875	3.35702957	0.00021183
resin system	35	115	0.001875	2.21404047	0.00549642
resin system	35	115	0.001875	2.06615758	0.00909429
brine	20	115	0.001875	3.66051692	0.00044579
brine	20	115	0.001875	3.47314647	0.00113942
resin system	20	115	0.001875	2.32742061	0.0003878
resin system	20	115	0.001875	1.76306118	0.00061863
brine	10	115	0.001875	2.75777643	0.0004665
brine	10	115	0.001875	2.7453632	0.00055122
resin system	10	115	0.001875	1.83695375	0.00043607
brine	5	115	0.001875	2.42721467	0.0005569
brine	5	115	0.001875	2.1267573	0.00509595

Table 21: Summary of spurt loss and C_w values

As already observed during the testing with the KCl brine there is randomness in the spurt values resulting from the S875 particle size distribution within one specific test and the particle deposition on the disc surface. Although the cross linking of the S875 leads to the low C_w values (also in previous test with KCl brine) it is assumed that at 115°F the fluid loss additive is not fully hydrated and cross linked after about 2 minutes, which is the time the product is allowed to soak in brine respectively resin. The fact that the particles might not be fully hydrated and cross linked by the time the particles are deposited on the filter medium is what might cause the favourable behaviour of high spurt loss at this temperature.

Testing on the filter discs using S875 in the resin system showed a reduced spurt loss compared to the test performed with brine as carrier fluid. The fluid loss control ability on the 10 and 20 micron disc was similar as in the brine system. Only the tests on the 35 micron sand disc showed higher C_w values and early break of the seal (Figure 92 and Table 21).

Figure 94 shows one of the filter cakes built with S875 and the water based resin system. The white parts on or within the filter cake are resin particles. A fluid loss test in the HPHT cell using the resin at 115°F without any fluid loss material did not show any fluid loss control or flow impairment through the filter disc. But as Table 21 shows the spurt loss somehow decreased at least 1gal/ft² from testing S875 in 6% KCl brine to testing it in the water based resin system. It has been observed that when switching off the stirring device after spurt loss and filter cake built up resin particles will deposit on top of the filter cake due to gravity. This deposition should not influence the spurt loss because as long as filter cake built up is not finished the stir runs at 250rpm. Therefore it is not clear what actually causes the decrease in spurt loss compared to testing with the 6% KCl brine at the same conditions.



Figure 94: Filter cake built during HPHT fluid loss test in water based resin system

4.3 Testing in a sand consolidation chamber

For the actual sand consolidation tests a Teflon sleeve sand consolidation chamber is attached to the bottom cap of the HPHT cell body. The sand consolidation chamber is filled with a sand mixture resembling actual formations encountered in real life situations. This chapter describes the preparation of various sand pack permeabilities, fluid loss test using 6% KCl brine and sand consolidation tests using FDP-S867A and FDP-S867B as carrier fluid of S875.

4.3.1 Preparation of the sand packs

The sand packs used for this testing series are prepared as follows. A 100 mesh screen is put on the bottom of the Teflon sleeve sand consolidation chamber. In order to prevent the fines added to the sand pack from going through the screen 10 grams of Oklahoma # 1 sand are put on top of the screen. Then the actual sand pack is put in the chamber. A rod is used to tighten the sand pack and to mark its length which is needed to calculate the permeability of the pack. Instead of putting 15g Oklahoma # 1 followed by a 40 mesh screen on top of the sand pack it has been decided to add another 10g of the sand mixture. This 10g are consolidated by using the epoxy resin Expedite 225. The resin Expedite 225A is mixed with the hardener (Expedite 225B) 1:1 and 0.6ml of the mixture are added to the sand. After wetting the sand with Expedite 225 the sand is put at the top of the unconsolidated sand mixture in the consolidation chamber. Again the sand pack is tightened with a rod and the final length of the sand pack is recorded. The sand consolidation chamber is put in the oven to cure the resin. In the oven the resin will cure in a few hours but it will also cure at room temperature over night. As the sand consolidation chamber is tapered the average diameter of the

chamber has been calculated to get the filtration area. The filtration area of the cell is 4.928cm^2 and the length of the sand packs is about 11cm.

The most important part of the whole procedure is the preparation of the sand mixture which determines the permeability of the sand pack. The sand pack consists of Oklahoma # 1, WAC-9 crystalline silica also used as fluid loss additive and bentonite. To vary the permeability of the sand pack the amount of clay has to be varied. The table below shows the composition of the sand packs and gives the expected permeability range of the mixture.

	Mixture 1	Mixture 2	Mixture 3	Mixture 4
Permeability range	120 – 200 mD	300 – 500 mD	around 1000 mD	> 1000 mD
Oklahoma # 1	85%	88%	89%	89.5%
WAC-9 Additive	10%	10%	10%	10%
Bentonite	5%	2%	1%	0.5%
KCl brine	0.65 ml	0.65 ml	0.65 ml	0.65 ml

Table 22: Composition of sand packs of various permeability

4.3.2 Fluid loss tests

Before testing with the resin system 6% KCl brine was used to adjust a proper FDP-S875 concentration leading to a high spurt loss and a low C_w value. Considering that bentonite is added to the sand packs the KCl concentration has to be at least 6% in order to prevent swelling of the clay and reduction of permeability. Further the influence of permeability variation on the achieved spurt loss will be tested.

The first testing series was performed using sand mixture number 2. The estimated permeability of this mixture lies between 300 and 500mD. Before running test April8_T2 the permeability of the sand pack has been calculated using 6% KCl brine at 100°F with an estimated viscosity value of 0.76 cp. A pressure of 300psi was applied on the HPHT cell. There was no backpressure applied and atmospheric pressure has not been taken into account, therefore the differential pressure used for the calculation was 300 psi. The sand pack permeability for test April8_T2 was 375mD and for test April8_T3 it was 389mD. It has to be mentioned that these permeability numbers are more an estimation than an exact calculation. In fact it should be used mainly to get an idea of the sand pack permeability to be able to compare it to the permeability range of the consolidated and cured sand packs.

Figure 95 shows the effect of various FDP-S875 concentrations on spurt loss and C_w . For April7_T1 1g of S875 was put into the HPHT cell body, the mixture was stirred while the top cap was screwed on the cell and the hose was connected. This procedure takes about 1.5 minutes, so the fluid loss test starts about 2 minutes after S875 was poured into the brine. The cell temperature is adjusted to 115°F. Due to small temperature changes during the procedure the amount of S875 is poured in at 111 or 112°F. The same procedure will be used for all tests performed. The concentration used in the first test led to a spurt loss of 45.83ml resulting in $2.282\text{gal}/\text{ft}^2$. For the second test (April8_T1) the amount of product was reduced to 0.4g which gives a concentration of 0.001g/ml. This reduction results in a spurt loss of 101.3ml or $5.044\text{gal}/\text{ft}^2$. To confirm this result the test was repeated at the same concentration and it resulted in a spurt loss of 110ml or $5.478\text{gal}/\text{ft}^2$. As the filtering area of a Fann filter disc (25.108cm^2) is about 5 times larger than the filtering area of the sand pack (4.928cm^2) it was tested if the product concentration can be reduced further. Taking the 0.75g from previous tests on the sand disc about 0.15g of S875 should be enough to cover the surface area of the sand pack. Therefore test April8_T2 was performed to evaluate the sealing ability of 0.2g S875 in 400ml brine on the sand pack surface (0.0005g/ml). Before establishing the filter cake 160ml brine has been lost, but the seal only lasted 2.5 minutes. Within this testing series the relationship between product concentration and spurt loss can be seen very well in the figure and table below. During these tests the decrease of concentration showed increase in spurt loss. To evaluate the behaviour of S875 at various sand pack permeabilities a fluid loss additive concentration of 0.4g FDP-S875 will be used.

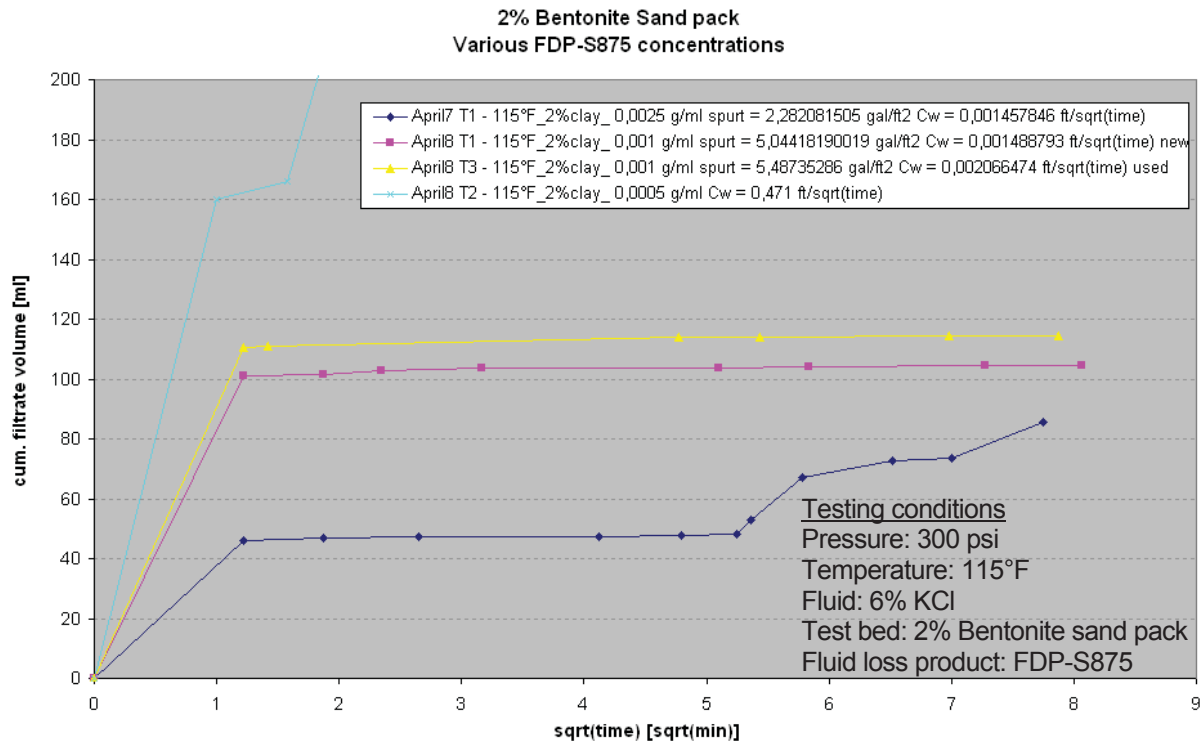


Figure 95: Variation of FDP-S875 concentration (2% bentonite sand pack)

Above figure shows the dependence of spurt loss and C_w on the concentration of FDP-S875. Reducing the S875 concentration by a factor 2.5 from 0.0025 to 0.001g/ml leads to an increase in spurt loss of a factor 2.2 respectively 2.4.

	concentration [g/ml]	spurt loss [gal/ft ²]	C_w [ft/sqrt(time)]
April7 T1	0.0025	2.28208151	0.00145785
April8 T1	0.001	5.0441819	0.00148879
April8 T3	0.001	5.48735286	0.00206647
April8 T2	0.0005		0.471

Table 23: Variation of FDP-S875 concentration (2% bentonite sand pack)

Table 23 does not include a spurt value in gal/ft² for test April8_T2 because the calculation would lead to a negative intersection on the y-axis. For the calculation of C_w in test April7_T1 only the data points recorded before the fluid breakthrough were used. As mentioned previously a fluid breakthrough will not cause a problem in an actual downhole situation. Referring to the tests with a product concentration of 0.0025 and 0.001g/ml the change in C_w is minor therefore indicating good fluid loss control behaviour for both concentrations. Test April8_T2 which was performed at a S875 concentration of 0.0005g/ml shows an unacceptable high C_w value.

In order to evaluate if the spurt loss obtained by using S875 as a diverting agent mainly depends on the permeability of the sand pack or on the product concentration further testing was done on sand packs containing various amounts of clay.

Figure 96 displays the fluid loss record over time for sand packs of various permeabilities. All four sand pack mixtures defined in the previous chapter have been prepared. This means the permeability ranges from about 0.12D by using 5% bentonite to above 1D by reducing the bentonite content to 0.5%. From the graphs it can be concluded that the spurt loss does not necessarily decrease with decreasing permeability. Test Apr9_T1 (the dark blue graph) was performed on the sand pack with the highest clay content (5%). From experience with these sand packs the permeability of a 5% bentonite sand pack lies between 120 and 200mD. Despite having the lowest permeability higher cumulative fluid loss could be observed on this sand pack than on the sand pack containing 2% clay (test Apr8_T1). For confirmation of this trend also test Apr8_T3 from

Figure 95 should be mentioned here because this test too showed lower spurt loss than the sand pack containing 5% clay. The pink graph, test Apr8_T1 (2% clay sand pack) had the lowest spurt value out of all four tests. The fluid loss test on the sand pack mixture with 1% clay resulted in the highest spurt loss value (6.06gal/ft²). It can be seen that before ending the test a slight increase in cumulative fluid loss occurs which indicates a breakthrough of fluid through the filter cake. No sealing filter cake could be created on the sand pack with 0.5% clay content. As filter disc permeabilities as high as 5.5D had been successfully sealed off with FDP-S875 and the estimated permeability of this pack lies slightly above 1D it should be no problem to create a seal. The initial fluid loss is very high and the curve shows that the fluid throughput slowed down a little. The high slope of the curve leads to a very low intersection on the ordinate and therefore to a low spurt value in gal/ft².

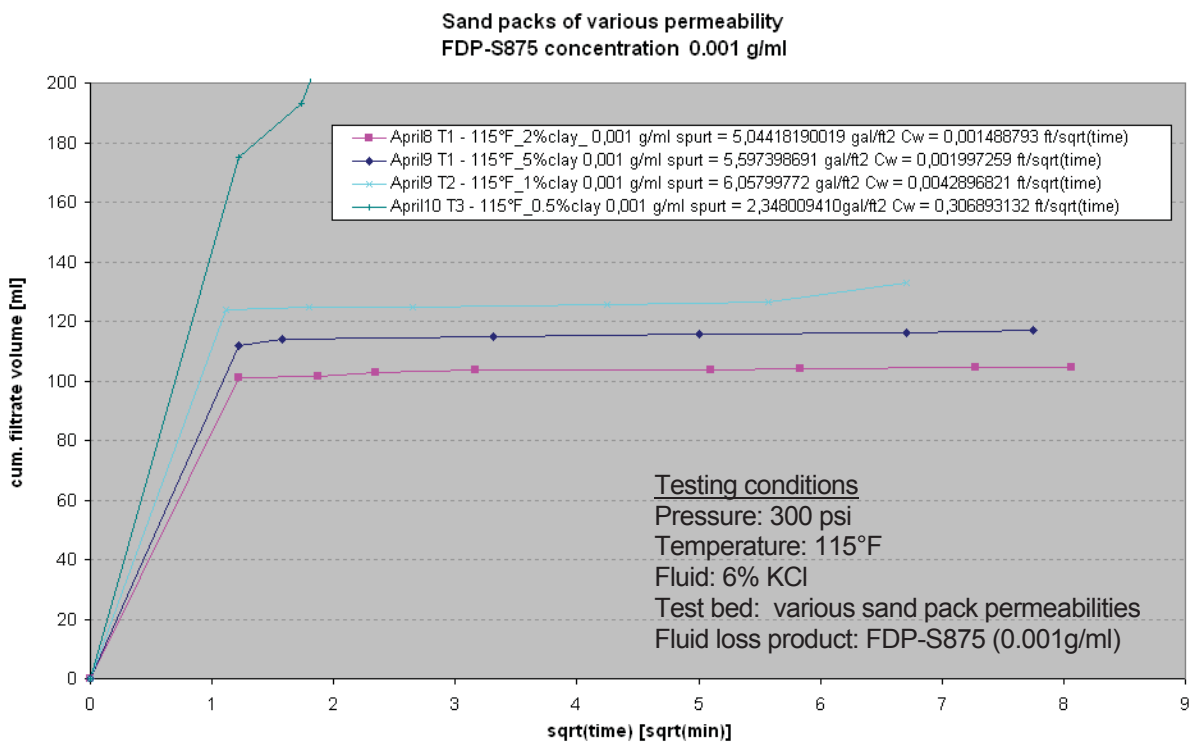


Figure 96: Variation of bentonite content in sand pack (0.001g/ml FDP-S875)

Table 24 summarizes the spurt loss and wall-building fluid loss coefficient values of the tests plotted in Figure 96.

	concentration [g/ml]	clay content [%]	spurt loss [gal/ft ²]	C _w [ft/sqrt(time)]
April10 T3	0.001	0.5	2.348	0.3069
April9 T2	0.001	1	6.058	0.0043
April8 T1	0.001	2	5.044	0.0015
April9 T1	0.001	5	5.597	0.0019

Table 24: Variation of bentonite content in sand pack (0.001g/ml FDP-S875)

4.3.3 Consolidation tests

After using previous tests to define a FDP-S875 concentration suitable to seal off the surface area of the sand pack the carrier fluid has been changed to the water based resin system FDP-S867A and B. Before injecting the resin the sand pack was saturated with brine and its permeability was estimated. The permeability values will be considered in chapter 4.4.

The first test (Apr17_T1) was performed on a sand pack containing 2% bentonite. No fluid loss additive has been added for this test. The objective was to figure out if the resin system itself would impair fluid flow through the sand pack. The resin has been poured in the HPHT cell body and the stirring device was not switched on and after applying 300psi pressure the discharge valve had been opened and fluid loss was recorded. The resulting curve can be seen in the diagram below (light green graph). Clearly in case of not keeping the resin system stirred gravity causes resin particles to fall out and act as fluid loss control additives. This does not explain the reduced spurt loss experienced when testing on the sand discs because the stirring continued until spurt loss has been finished. So in further testing the stir is set to 250rpm and stirring continues until the end of the fluid loss test. For comparison test Apr20_T1 has been performed. It contains the same amount of clay and also no FDP-S875 was added to the resin. The only difference is the continuous stirring throughout the entire fluid loss test. It can be seen that within 3.5 minutes 46ml of resin are filtrated through the sand pack. The graph is a straight line no reduction of flow rate can be observed. In case of no stirring on the other hand the curve is similar to actual fluid loss curves with added FDP-S875. In test Apr17_T1 it took 20 minutes for 44ml of resin to pass through the sand pack. Both tests have been stopped after a fluid throughput of about 43ml.

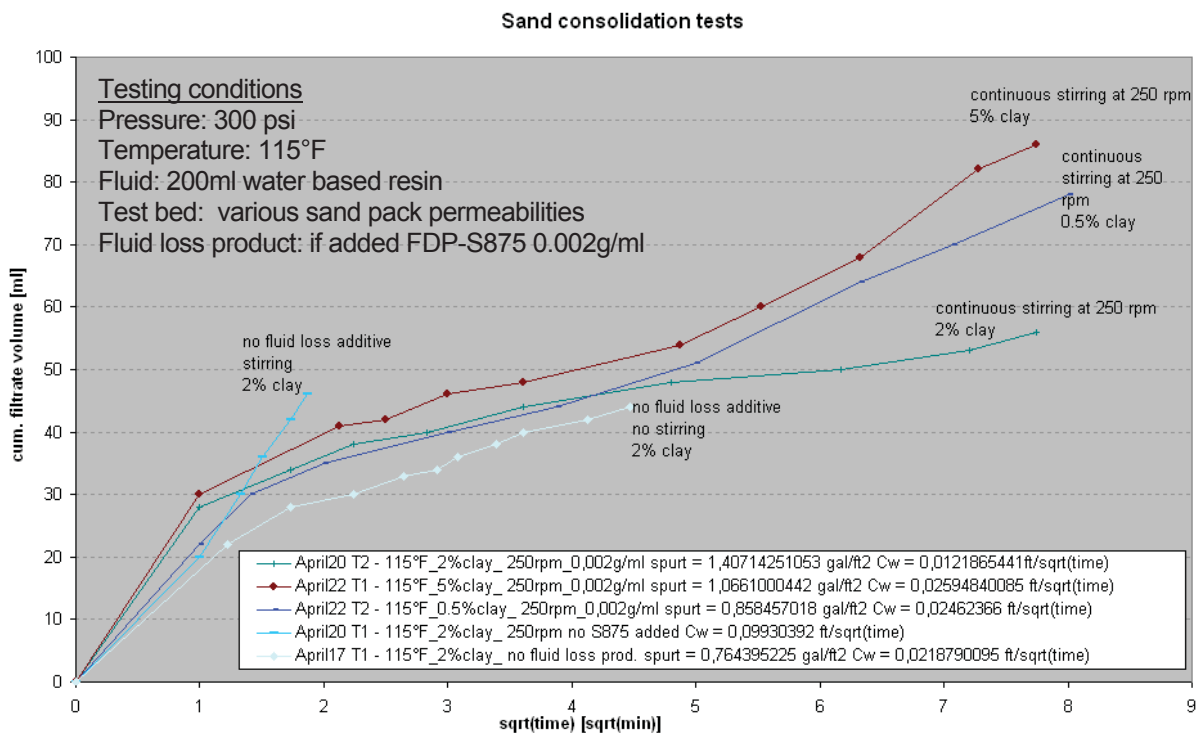


Figure 97: Sand consolidation tests

Test Apr20_T2 was performed on a sand pack with clay content of 2%. The amount of FDP-S875 was 0.4g this resulted in a concentration of 0.002g/ml in 200ml resin system. During the test the resin system was stirred at 250rpm to prevent the resin particles from falling and act as an additional fluid loss control agent. The other two tests Apr22_T1 and Apr22_T2 were performed at the same conditions but on sand packs of different permeabilities. As in previous testing with the brine the sand pack with 2% clay showed the lowest cumulative fluid loss. Other than in the previous testing the lowest cumulative losses did not lead to the lowest spurt value. As it can be seen in the table below the highest spurt value (1.47071gal/ft²) was achieved. The reason is the better fluid loss control over time compared to the other two tests where S875 was used. In case of the 5% and 0.5% bentonite sand packs the slope of the created trendline is higher and therefore lower spurt values are obtained. A summary can be found in Table 25. Compared to the tests with the 6% KCl brine as carrier fluid the (Table 24) the spurt values by using the resin system decreased from above 5gal/ft² down to about 1gal/ft². As during the entire tests with the resin

system the stir has been working on 250rpm the reason which results in reduction of spurt loss can not be settling due to gravity.

	concentration [g/ml]	clay content [%]	spurt loss [gal/ft ²]	C _w [ft/sqrt(time)]
April17 T1	-	2	0.7644	0.02188
April20 T1	-	2	-	0.00993
April20 T2	0.001	2	1.4071	0.01219
April22 T1	0.001	5	1.0661	0.02594
April22 T2	0.001	0.5	0.8584	0.02462

Table 25: Summary of consolidation tests

The samples Apr17_T1, Apr20_T1 and Apr20_T2 have been cured for 72 hours at 180°F. The samples Apr22_T1 and Apr_T2 have been cured for 120 hours. After the curing period the regain permeability has been tested. Subsequently the samples were taken out of the Teflon sleeve chamber. In some cases the bottom of the sand pack was not entirely consolidated.

These tests did not show a spurt loss and sealing performance as good as those using the brine system as carrier fluid for the FDP-S875. The consolidated samples are used to define a strength range of the consolidated packs and get an idea of the resin distribution in the pack via loss on ignition tests. It is recommended to perform further testing on sand packs in order to figure out if the created almost perfect fluid loss curves from previous testing can also be created with the water based resin system.

4.4 Regain Permeability Tests

For Apr20_T2, Apr22_T1 and Apr22_T2 tests have been performed to get an idea of the percentage of permeability regained after curing of resin and acidizing the guar filter cake.

The initial permeability of the sand packs were determined when the sand pack had been saturated with brine. The tests were performed at 100°F and the viscosity value for the 6%KCl brine was 0.76cp. The surface area of those sand packs is 4.928cm².The pressure applied on the HPHT cell was used as pressure differential, as mentioned before no backpressure was applied. The objective was to confirm that the permeability value is within the defined range of each mixture and to get values for comparison with the regain permeability tests. The calculation was performed in an Excel spreadsheet.

$$k = -\frac{q\mu L}{A\Delta p}$$

- μ = fluid viscosity [cp]
- q = fluid flow rate [ml/sec], [cm³/sec]
- k = permeability [D]
- A = cross-sectional area [cm²]
- dp = pressure drop [atm]
- dL = flow length [cm]

For Apr20_T2 the following results were obtained. The 2% clay sand pack is estimated to have permeabilities between 300 to 500mD. The length of this specific sand pack was 11.13cm and the flow rate recorded at a pressure difference of 100psi was 2.333ml/sec (70ml in 30 sec). With above defined brine viscosity and surface area the sand pack permeability was calculated to be 588mD. For the determination of permeability and regain permeability the HPHT cell body was filled with 400ml brine, the fluid output in 60 seconds or for the higher permeable packs in 30 seconds were recorded, an average value out of all records is taken and the flow rate per second is calculated.

Concerning the test of the sand pack regain permeability it has to be mentioned that these tests could not be performed as wanted. The ideal way would be to put heated 15% HCl in the HPHT cell body and apply pressure in order to bring the HCl on top of the filter cake in the sand consolidation chamber attached at the HPHT cell discharge valve. As the HPHT cell is manufactured out of 316 stainless steel it can not be used for acid testing. The acid tests could induce chloride stress cracking in the HPHT cell. Regrading the safe work environment that should be provided for further work with this cell no HCl was put in the HPHT cell. For testing with HCl it is necessary to fabricate cells from Monel or Hastelloy. This safety issue is the reason why the regain permeability tests are performed by pouring about 6ml of 150°F HCl on top of the filter cake (the HCl touches only the walls of the Teflon sleeve inside the metal sand consolidation chamber). The big disadvantages are that the HCl cools down to room temperature very quickly and no pressure can be applied. Therefore it has to be kept in mind that the results shown subsequently can be way better by soaking the filter cake in hot HCl and applying pressure.

For test Apr20_T2 acid has been on the filter cake top for 3.5 hours, it was reheated to 150°F three times. The flow rate obtained when the sand consolidation chamber was attached to the HPHT cell and 6%KCl at 100°F were filtered through the consolidated pack by applying a pressure of 100psi. A resulting flow rate of 0.1667ml/sec was recorded. With the unchanged values of length and surface area the permeability of the sand pack was 70md or 0.07D. This gives a regain permeability of only 11.90%.

Test Apr22_T1 is the sand pack with the highest clay content (5%). The permeability has been determined with 100°F warm 6%KCl brine. The surface area of the sand pack is the same; this specific pack had a length of 11.55cm. The pressure applied during the permeability determination was 60 psi and a flow rate of 0.5333ml/sec was recorded (32ml in 60sec). This leads to a permeability value of 232mD for this sand pack. The sand pack has been acidized after curing as described for test Apr20_T2. The first regain permeability tests were performed at 60psi, but the flow rates were really low and would result in a regain permeability of about 4%. As no pressure has been applied during the acid soak the HPHT cell was filled with brine and a pressure of 300psi has been applied in order to open channels created by the acid soak. After performing brine filtrations at higher pressure another trial of regain permeability measurement was performed. The pressure has been decreased to 100psi and fluid output was recorded for 60 seconds. The cell was filled with 400ml brine and the flow rates were recorded for about ten minutes. Then the brine has been replaced and another test was performed. The first test resulted in an average fluid output of 20ml and the second one in 14ml per 60 seconds which are flow rates of 0.33 respectively 0.233ml/sec. The regain permeability calculated was 87mD for the higher and 61mD for the lower flow rate. The percentage of regained permeability lies between 26.3 and 37.5%.

Test Apr22_T2 is the highest permeable sand pack with bentonite content of 0.5%. The length of this sand pack was also 11.55cm and at a pressure of 60psi 68ml fluid throughput in 30 seconds was recorded. This leads to a flow rate of 2.2667ml/sec, which gives an initial sand pack permeability of 988mD. The same procedure of acid soak was performed on this filter cake. The permeability after the acid treatment was 523mD which gives a regain permeability of 52.9%.

When looking at these regain permeability values two things have to be kept in mind. The first one is the testing procedure which could be improved a lot with new equipment suited for acid treatments. And the second is that the C_w values of all three samples were around 0.02 ft/sqrt(min) which means that compared to tests with brine as carrier fluid the sealing ability is worse. It would be interesting for future tests to analyse the regain permeability of a cake which almost shut off the fluid filtration.

4.5 Tensile Strength and L.O.I. Tests

The average diameter of the consolidated sand pack is 1inch. The Teflon sleeve chamber is tapered, its diameter on top is larger than on bottom. Below figure shows a consolidated sand pack already removed from the Teflon sleeve. It can be seen that in this specific case the sand on bottom has not been consolidated. This picture shows the sample created from test Apr17_T1, which means no S875 has been added and the resin has not been stirred during testing. As the resin

plugged the sand pack surface and reduced resin throughput not enough resin reached the bottom of the sand pack to consolidate it.



Figure 98: Consolidated sand pack

It has been decided to perform a tensile strength test instead of a compressive strength test. The strength test apparatus offers beside other tests the Barton test (compressive strength test) and the Brazilian strength test (tensile strength test). For the Barton test samples with a length of 1.5 times the core diameter are needed. As the samples created in the sand consolidation chamber are about 4.3in long (if consolidated to the bottom) not even 3 samples could be cut out of one core. Therefore samples for the Brazilian strength test were prepared. The length of the samples should be about 0.5in. This way it was possible to create up to seven samples out of one core. By testing the core strength in a distance of 0.5in a good idea of the resin distribution in the core will be obtained. Figure 99 shows the dimension of the samples used for the strength tests and the position in which the sample is placed in the test apparatus.

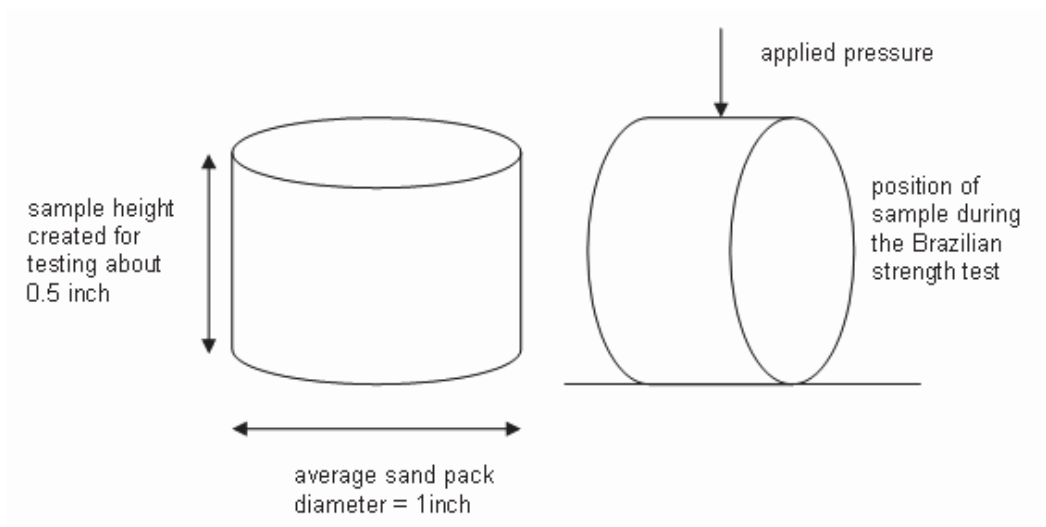


Figure 99: Sample preparation for the Brazilian strength test

In below figures samples taken from the sand pack with the 0.5% and 5% clay content are shown.



Figure 100: 0.5% clay – S875 filter cake and previously consolidated top plug



Figure 101: 0.5% clay – top 0.5inch of the consolidated sample



Figure 102: 5% clay – top 0.5inch of the consolidated sample



Figure 103: 5% clay – bottom 1inch of the consolidated sample

Comparing Figure 101 and 102 which both show the top 0.5in of the consolidated sand pack (0.5in below the Expedite 225 consolidated part) it can be seen that in Figure 102 the center of the core is less consolidated than the annulus. Parts of the sand even could be scraped away; Figure 101 on the other hand shows a uniformly consolidated top sample. In case of the 5% clay sample it can be seen that the tendency of consolidating the outer boundary and leaving a softer center decreased in the samples on bottom of the core.

The following diagram summarizes the results of the strength tests on the created core consolidated samples. On the x-axis the averaged lengths of all cut samples is summed up to one cumulative core. This way it is possible to compare the strength of the cores. The first thing recognized is that the resin strength forms a wave in two out of the five consolidated cores. The yellow data set (Apr20_T2) shows that within the first 0.5in below the Expedite plug less tensile strength is provided by the resin than in the second 0.5in below the top plug. The same is true for the first data set (Apr17_T1), but the peak of tensile strength can be found 0.5in below the one of the Apr20_T2. The other samples showed a decrease in tensile strength with core length. Detailed strength values for each sample can be found in Appendix B.

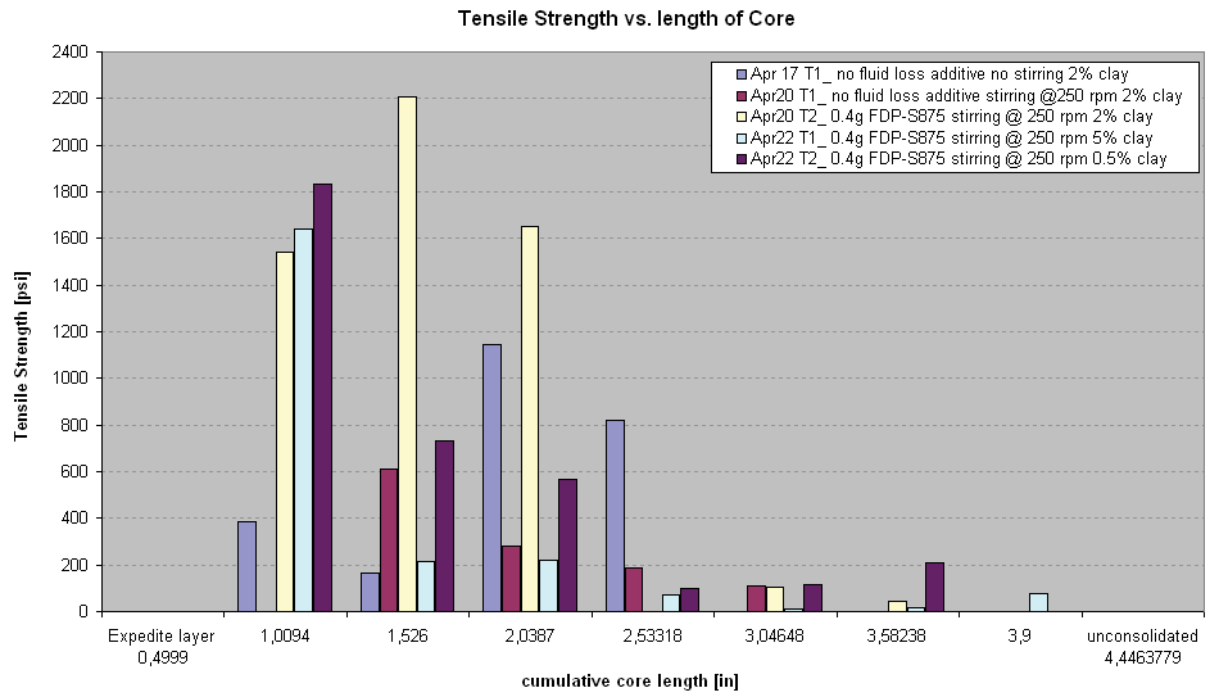


Figure 104: Tensile strength of consolidated samples vs. core length

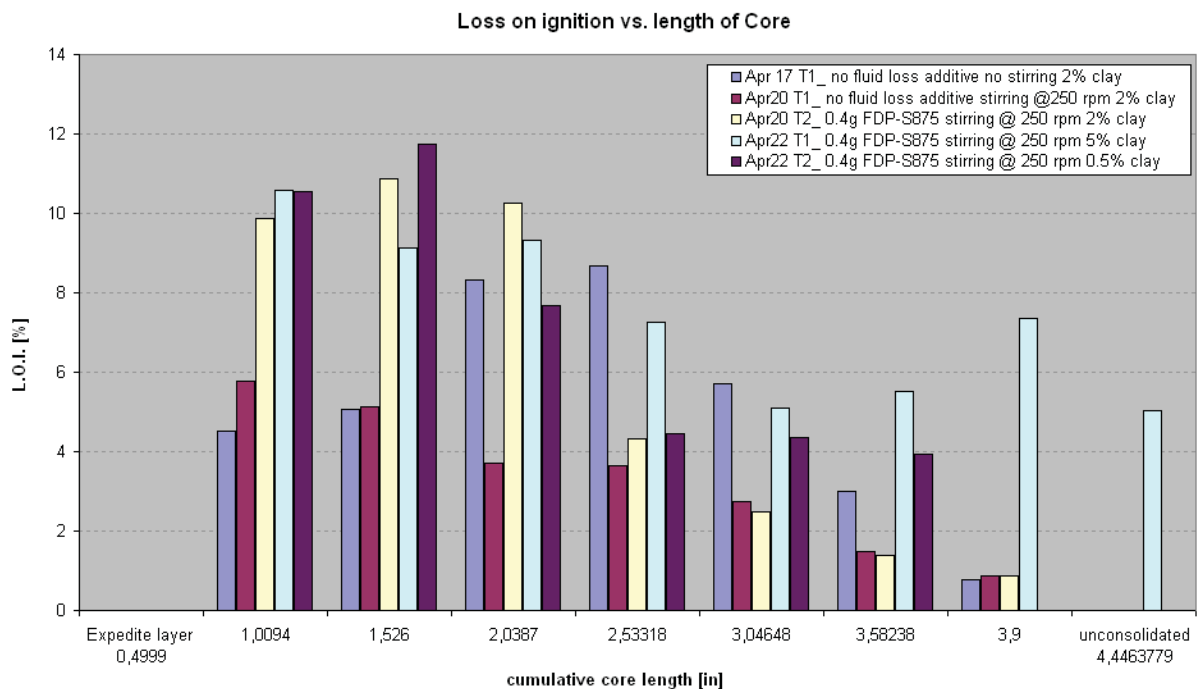


Figure 105: Loss on ignition of consolidated samples vs. core length

In order to define pore volumes or to get an idea of the amount of resin transported to a certain point within the sand pack a loss on ignition test (L.O.I. test) has been performed by the analytical department. To perform this test the samples are taken and weighed (the samples are broken after the strength test but this does not matter for the L.O.I. because also unconsolidated material from the bottom of the sand consolidation chamber was handed in for analysis), burned in an oven at 1000°C and weighed again. The resin will be burned after taking the sample out of the oven. The weight loss defines the amount of resin in the sample, the data submitted is the % weight lost on ignition of the samples. Figure 105 shows a comparison of the values obtained in the L.O.I. testing.

In order to get a better idea of the relationship between the L.O.I. and the tensile strength diagrams were created which plot the results of both tests for the three sand packs of various permeabilities.

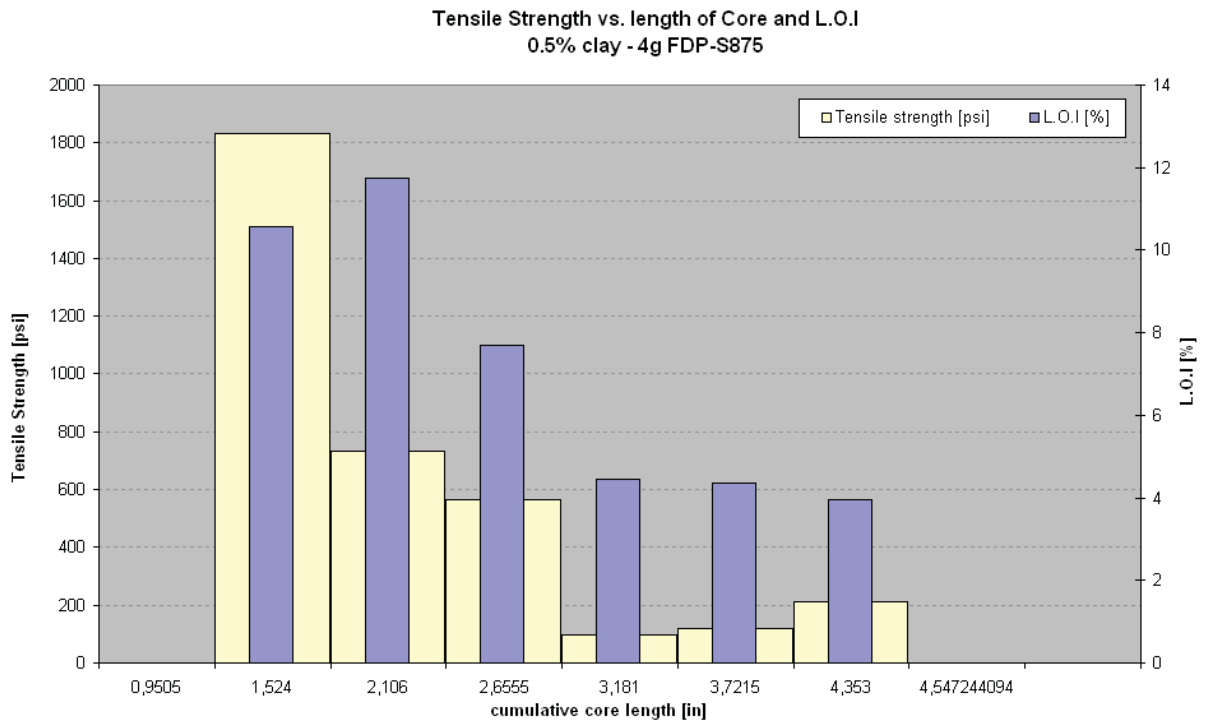


Figure 106: Tensile strength and LOI vs. length of core – 0.5% clay sand pack

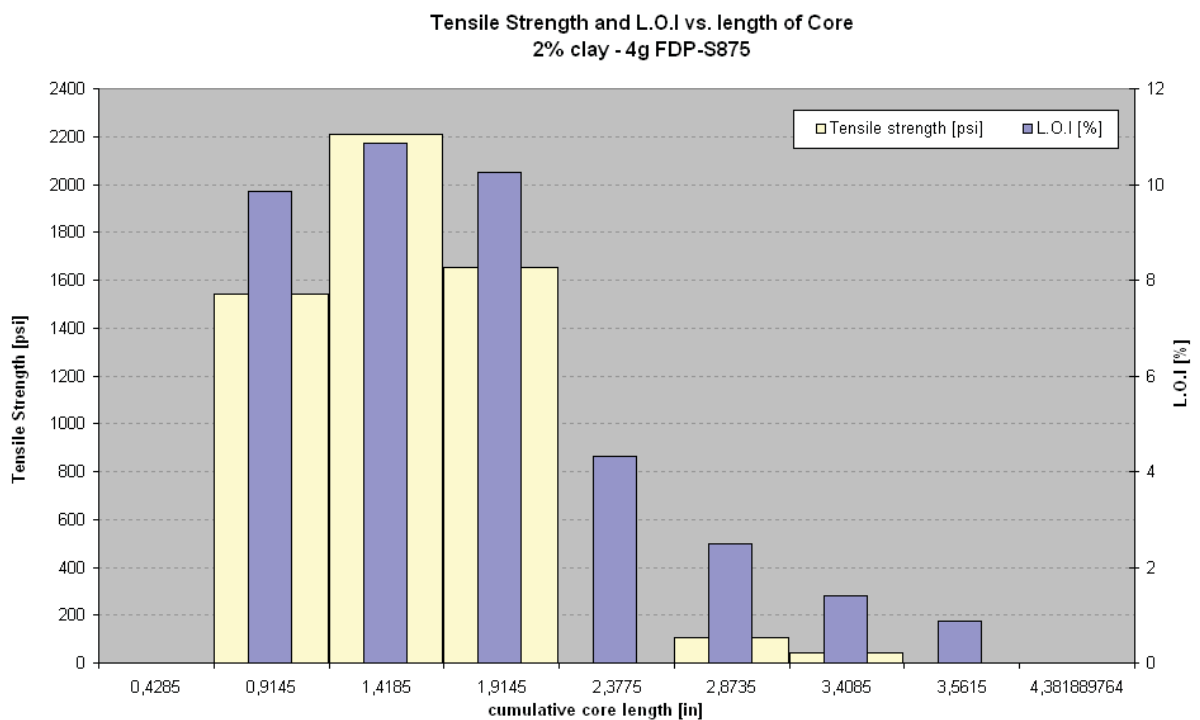


Figure 107: Tensile strength and LOI vs. length of core – 2% clay sand pack

Figure 106 shows that the tensile strength within this consolidated sample (Apr22_T2) decreases from top to a core length of about 3in. From this point on the strength showed a slight increase to the bottom of the core. After a slight increase in loss on ignition at a core length of 2in the L.O.I. decreased to the bottom of the core.

The sand pack having a clay content of 2% (Figure 107) showed a wave in the tensile strength distribution over the core length. Other than in Figure 106 and 108 in this core the tensile strength can be well related to the amount of resin in the parts of the core. Also the L.O.I distribution shows a curve with its peak 1.4in below the filter cake surface area.

In Figure 108 the test results obtained from the sand pack with the highest clay content are shown. The samples shown in Figure 102 and 103 belong to the values below. As described before these samples had a rather soft core and a well consolidated annulus. The tensile strength of the core decreases from top to bottom. The amount of resin in the core at a cumulative core length from 1.2 to 1.87in lies above 9%. But according to the strength test values this amount of resin could not create high strength in this section of the core.

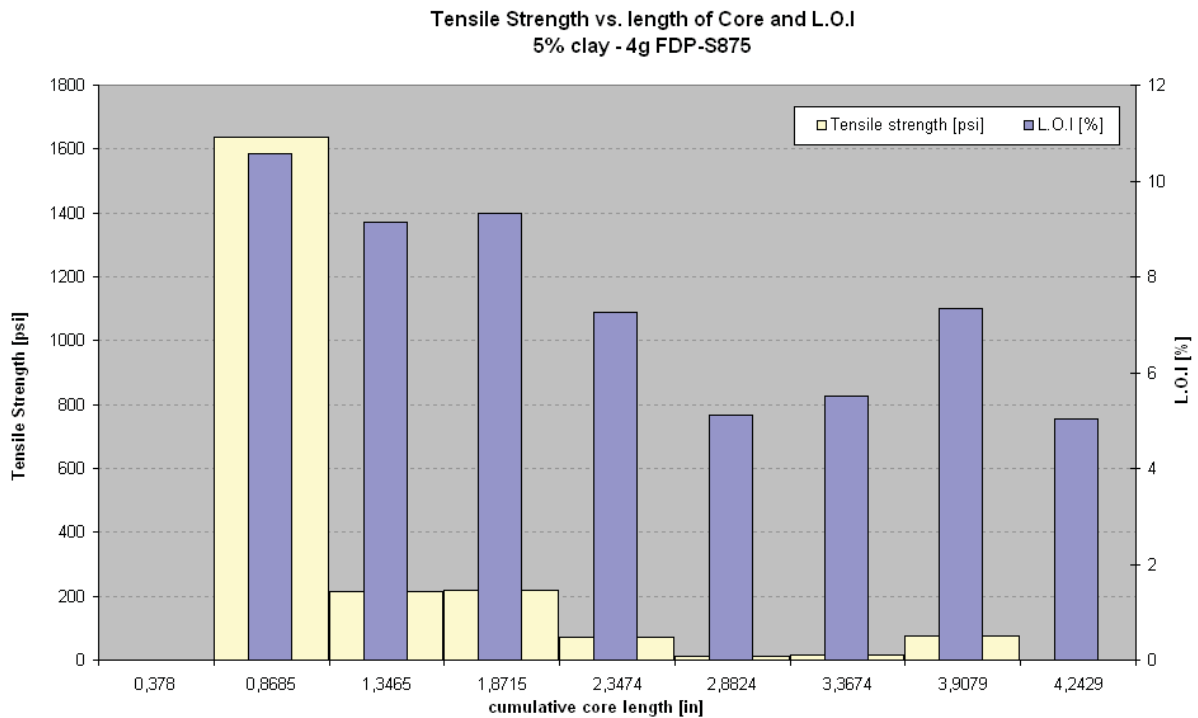


Figure 108: Tensile strength and LOI vs. length of core – 5% clay sand pack

Tables showing the results of the Brazilian strength test and the loss on ignition tests are shown in Appendix B. Also

Conclusion

For the fluid loss additive FDP-S875 a combination of temperature and soaking time has been found which favours the concept of diversion defined by high spurt losses and build up of very good sealing abilities. This promising behaviour and the theory of a fluid loss product with a hard center and a soft coating on the outside will lead to continuous testing on FDP-S875 and also lead to research for this behavior in other products.

At temperatures of 150°F and above the sealing ability of FDP-S875 is extraordinary well, just the spurt loss is not as high as desired for this diversion concept. At room temperatures the product acts like a bridging agent and in order to create filter cakes with minimum permeability the particles have to be manufactured according to particle size distributions suitable for a range of formation pore sizes. Increasing the product soaking time at room temperature changes the texture of the filter cake from brittle pack to a gel. The best results could be obtained from testing the product at 115°F and giving the particles about 2 minutes time to soak in brine. At this condition the high spurt loss could be combined with extraordinary well sealing abilities.

Further the spurt losses obtained from testing FDP-S875 are not highly dependent on the permeability variation of the formation. The ideal conditions of total independence of spurt loss from permeability could not be seen. In general the tests showed that in case of lower permeability difference (between 5 and 10 micron sand disc the permeability increases by a factor 1.27) the increase in spurt loss per unit area lies in the same range (1.26-fold increase in spurt loss from 5 to 10 micron disc). The results get better at higher permeability variation. While the permeability from 5 to 35 micron filter discs increase by a factor 7.33 the spurt loss increases only by a factor 1.54. These values confirm that FDP-S875 allows high spurt losses also at lower permeable formations and therefore favours uniform distribution of formation treatment fluids.

Another thing observed from testing of FDP-S875 is that spurt loss does not necessarily increase with the reduction of product concentration in the carrier fluid.

The results described were obtained by using KCl brine as carrier fluid for the fluid loss additive. Switching from brine to water based resin as carrier fluid showed a decrease in spurt loss values although the tests on the sand disc showed that the resin system itself will not act as a fluid loss control agent as long as not heated up to about 175°F. Again the spurt loss independence on permeability showed good results.

The results obtained from testing in the sand consolidation chamber showed that FDP-S875 does not decrease the strength of the consolidated sand packs compared to those consolidated without using FDP-S875 as fluid loss additive. Further tests should be done to improve the filter cake performance in the sand consolidation chamber. During the consolidation tests the filter cakes showed fluid loss control but compared to the cakes created in the brine systems the sealing ability was not good. Although the resin system is stirred during the entire test and under this condition it did not act as a fluid loss additive itself it somehow seems to interact with FDP-S875. This interaction showed lower spurt loss on the filter discs but still good C_w values. In case of sand consolidation tests both spurt loss and sealing ability were worse than in the brine system.

For further testing it is recommended to check the purity of the KCl solutions in order to confirm its 6% KCl content. The regain permeability tests should be conducted in suitable HPHT cells in order to be able to apply pressure during the HCl soak. This might lead to higher regain permeability values. Only linear flow tests have been performed so far, if time and resources allow radial flow tests with the FDP-S875 product this option should be considered.

Abbreviations

H	= thickness of the zone
ΔH	= vertical compaction
Δe	= change in void ratio (void ratio = volume of voids / volume of solids)
e_0	= original void ratio
Φ	= decimal porosity
EOR	= Enhanced oil recovery
v_t	= particle settling velocity
d_p	= particle diameter
ρ_p	= particle density
g	= gravity constant
ρ_f	= fluid density
μ	= fluid viscosity
q	= fluid flow rate
k	= permeability
A	= cross-sectional area
dp/dL	= pressure drop per unit length
HPHT	= high pressure high temperature
K_w or C_w	= wall building fluid loss coefficient
m	= slope of fluid loss curve, plotting cumulative filtrate volume from a filter press vs. the square root of flow time
A	= cross-sectional area of test media through which flow takes place
r	= wellbore radius
R	= penetration radius
D	= penetration depth ($D = R-r$)
Φ	= formation porosity
MSDS	= Material Safety Data Sheet
L.O.I.	= Loss on ignition
IFT	= Interfacial tension

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Appendix A: Calculations

In below tables the spurt values for 7 and 8 inch wellbores in 30% porous formation are given.

Penetration radius [ft]	penetration depth [ft]	spurt [gal/ft ²]
0.6	0.30833333	1.05761857
0.7	0.40833333	1.55771
0.8	0.50833333	2.13473857
0.9	0.60833333	2.78870429
1	0.70833333	3.51960714
1.1	0.80833333	4.32744714
1.2	0.90833333	5.21222429
1.3	1.00833333	6.17393857
1.4	1.10833333	7.21259
1.5	1.20833333	8.32817857
1.6	1.30833333	9.52070429
1.7	1.40833333	10.7901671
1.8	1.50833333	12.1365671
1.9	1.60833333	13.5599043
2	1.70833333	15.0601786

Table 26: Required spurt loss (wellbore diameter 7 [in], $\Phi=30\%$)

Penetration radius [ft]	penetration depth [ft]	spurt [gal/ft ²]
0.6	0.26666667	0.83776
0.7	0.36666667	1.27534
0.8	0.46666667	1.78024
0.9	0.56666667	2.35246
1	0.66666667	2.992
1.1	0.76666667	3.69886
1.2	0.86666667	4.47304
1.3	0.96666667	5.31454
1.4	1.06666667	6.22336
1.5	1.16666667	7.1995
1.6	1.26666667	8.24296
1.7	1.36666667	9.35374
1.8	1.46666667	10.53184
1.9	1.56666667	11.77726
2	1.66666667	13.09

Table 27: Required spurt loss (wellbore diameter 8 [in], $\Phi=30\%$)

In below figure and tables the spurt values for 5, 6, 7 and 8 inch wellbores in 25% porous formation are given.

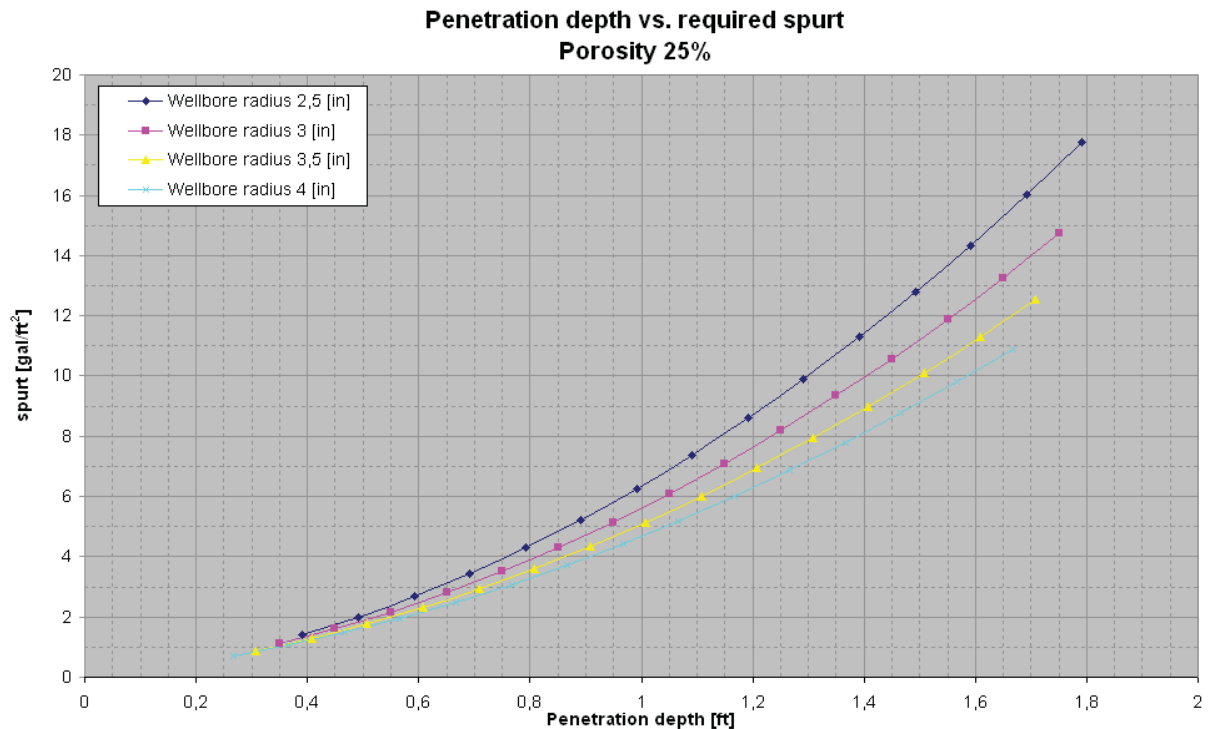


Figure 109: Penetration depth vs. required spurt loss (formation porosity 25%)

Penetration radius [ft]	penetration depth [ft]	spurt [gal/ft ²]
0.6	0.391666667	1.42088833
0.7	0.491666667	2.00432833
0.8	0.591666667	2.67752833
0.9	0.691666667	3.44048833
1	0.791666667	4.29320833
1.1	0.891666667	5.23568833
1.2	0.991666667	6.26792833
1.3	1.091666667	7.38992833
1.4	1.191666667	8.60168833
1.5	1.291666667	9.90320833
1.6	1.391666667	11.2944883
1.7	1.491666667	12.7755283
1.8	1.591666667	14.3463283
1.9	1.691666667	16.0068883
2	1.791666667	17.7572083

Table 28: Required spurt loss (wellbore diameter 5 [in], $\Phi=25\%$)

Penetration radius [ft]	penetration depth [ft]	spurt [gal/ft ²]
0.6	0.35	1.11265
0.7	0.45	1.59885
0.8	0.55	2.15985
0.9	0.65	2.79565
1	0.75	3.50625
1.1	0.85	4.29165
1.2	0.95	5.15185
1.3	1.05	6.08685
1.4	1.15	7.09665
1.5	1.25	8.18125
1.6	1.35	9.34065
1.7	1.45	10.57485
1.8	1.55	11.88385
1.9	1.65	13.26765
2	1.75	14.72625

Table 29: Required spurt loss (wellbore diameter 6 [in], $\Phi=25\%$)

Penetration radius [ft]	penetration depth [ft]	spurt [gal/ft ²]
0.6	0.308333333	0.88134881
0.7	0.408333333	1.29809167
0.8	0.508333333	1.77894881
0.9	0.608333333	2.32392024
1	0.708333333	2.93300595
1.1	0.808333333	3.60620595
1.2	0.908333333	4.34352024
1.3	1.008333333	5.14494881
1.4	1.108333333	6.01049167
1.5	1.208333333	6.94014881
1.6	1.308333333	7.93392024
1.7	1.408333333	8.99180595
1.8	1.508333333	10.113806
1.9	1.608333333	11.2999202
2	1.708333333	12.5501488

Table 30: Required spurt loss (wellbore diameter 7 [in], $\Phi=25\%$)

Penetration radius [ft]	penetration depth [ft]	spurt [gal/ft ²]
0.6	0.266666667	0.69813333
0.7	0.366666667	1.06278333
0.8	0.466666667	1.48353333
0.9	0.566666667	1.96038333
1	0.666666667	2.49333333
1.1	0.766666667	3.08238333
1.2	0.866666667	3.72753333
1.3	0.966666667	4.42878333
1.4	1.066666667	5.18613333
1.5	1.166666667	5.99958333
1.6	1.266666667	6.86913333
1.7	1.366666667	7.79478333
1.8	1.466666667	8.77653333
1.9	1.566666667	9.81438333
2	1.666666667	10.9083333

Table 31: Required spurt loss (wellbore diameter 8 [in], $\Phi=25\%$)

In below figure and tables the spurt values for 5, 6, 7 and 8 inch wellbores in 20% porous formation are given.

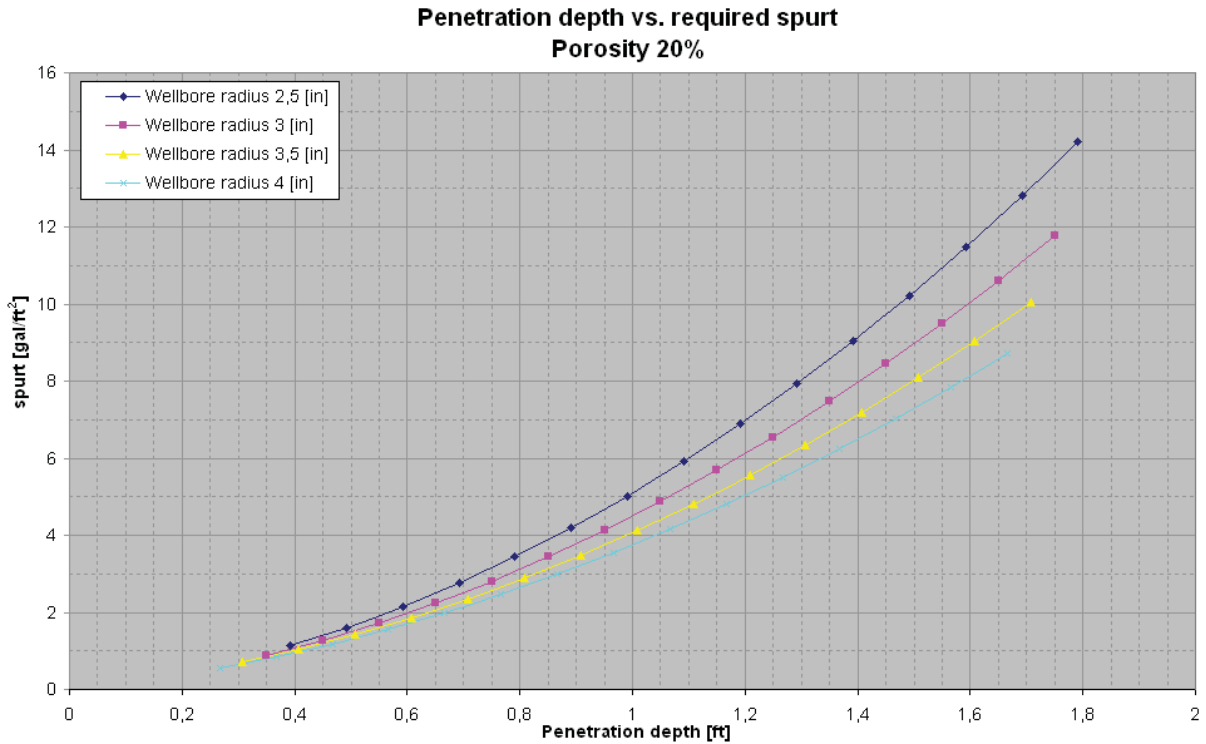


Figure 110: Penetration depth vs. required spurt loss (formation porosity 20%)

Penetration radius [ft]	penetration depth [ft]	spurt [gal/ft ²]
0.6	0.39166667	1.13671067
0.7	0.49166667	1.60346267
0.8	0.59166667	2.14202267
0.9	0.69166667	2.75239067
1	0.79166667	3.43456667
1.1	0.89166667	4.18855067
1.2	0.99166667	5.01434267
1.3	1.09166667	5.91194267
1.4	1.19166667	6.88135067
1.5	1.29166667	7.92256667
1.6	1.39166667	9.03559067
1.7	1.49166667	10.2204227
1.8	1.59166667	11.4770627
1.9	1.69166667	12.8055107
2	1.79166667	14.2057667

Table 32: Required spurt loss (wellbore diameter 5 [in], $\Phi=20\%$)

Penetration radius [ft]	penetration depth [ft]	spurt [gal/ft ²]
0.6	0.35	0.89012
0.7	0.45	1.27908
0.8	0.55	1.72788
0.9	0.65	2.23652
1	0.75	2.805
1.1	0.85	3.43332
1.2	0.95	4.12148
1.3	1.05	4.86948
1.4	1.15	5.67732
1.5	1.25	6.545
1.6	1.35	7.47252
1.7	1.45	8.45988
1.8	1.55	9.50708
1.9	1.65	10.61412
2	1.75	11.781

Table 33: Required spurt loss (wellbore diameter 6 [in], $\Phi=20\%$)

Penetration radius [ft]	penetration depth [ft]	spurt [gal/ft ²]
0.6	0.30833333	0.70507905
0.7	0.40833333	1.03847333
0.8	0.50833333	1.42315905
0.9	0.60833333	1.85913619
1	0.70833333	2.34640476
1.1	0.80833333	2.88496476
1.2	0.90833333	3.47481619
1.3	1.00833333	4.11595905
1.4	1.10833333	4.80839333
1.5	1.20833333	5.55211905
1.6	1.30833333	6.34713619
1.7	1.40833333	7.19344476
1.8	1.50833333	8.09104476
1.9	1.60833333	9.03993619
2	1.70833333	10.040119

Table 34: Required spurt loss (wellbore diameter 7 [in], $\Phi=20\%$)

Penetration radius [ft]	penetration depth [ft]	spurt [gal/ft ²]
0.6	0.26666667	0.55850667
0.7	0.36666667	0.85022667
0.8	0.46666667	1.18682667
0.9	0.56666667	1.56830667
1	0.66666667	1.99466667
1.1	0.76666667	2.46590667
1.2	0.86666667	2.98202667
1.3	0.96666667	3.54302667
1.4	1.06666667	4.14890667
1.5	1.16666667	4.79966667
1.6	1.26666667	5.49530667
1.7	1.36666667	6.23582667
1.8	1.46666667	7.02122667
1.9	1.56666667	7.85150667
2	1.66666667	8.72666667

Table 35: Required spurt loss (wellbore diameter 8 [in], $\Phi=20\%$)

Appendix B: Measurements

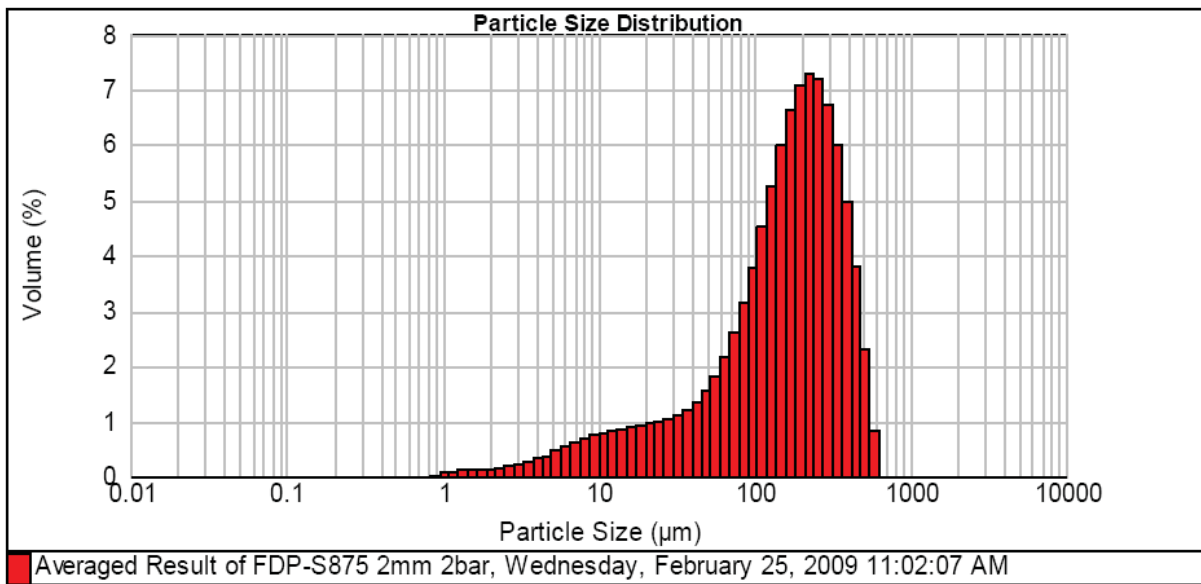


Figure 111: Particle size distribution of FDP-S875 cyclone mill grind (screen size 2 mm)

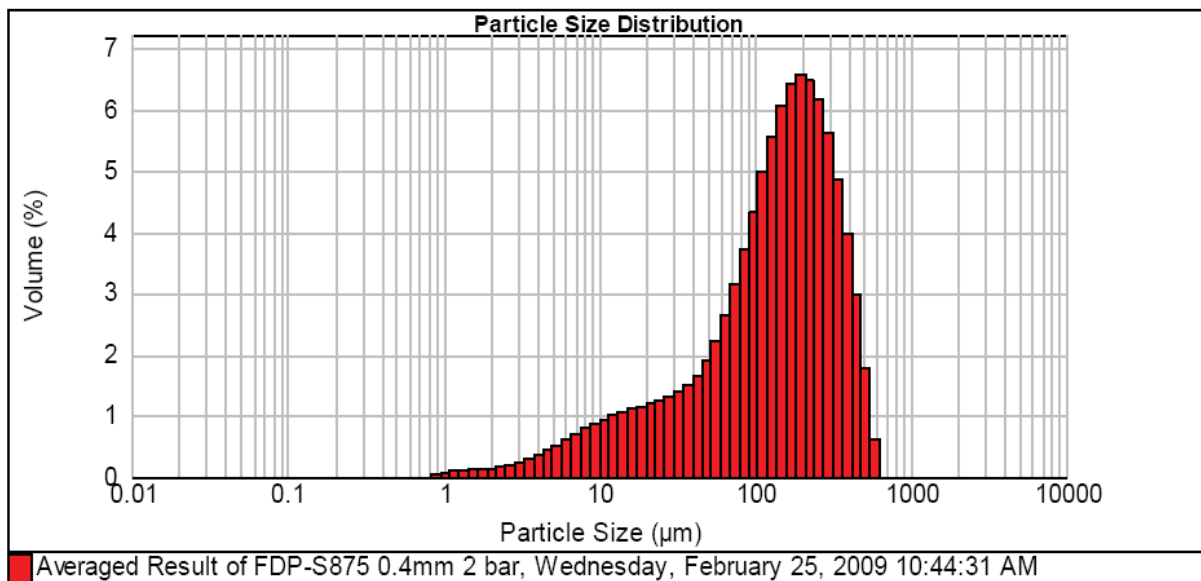


Figure 112: Particle size distribution of FDP-S875 cyclone mill grind (screen size 0.4 mm)

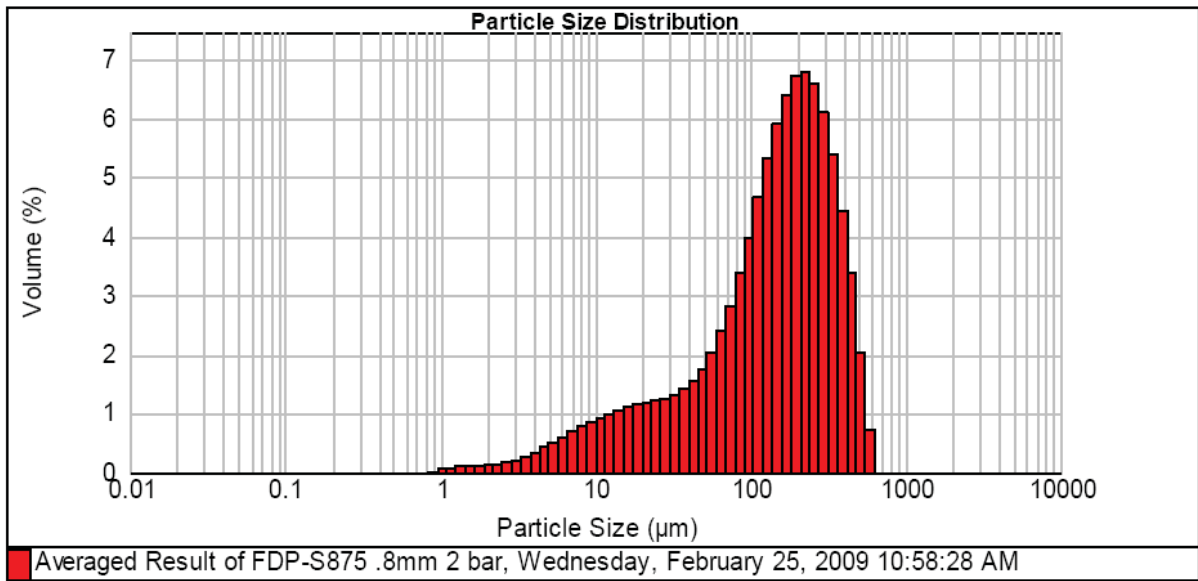


Figure 113: Particle size distribution of FDP-S875 cyclone mill grind (screen size 0.8 mm)