

# Experimental Study of Cement-Formation Bonding

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# HOVEDOPPGAVE/DIPLOMA THESIS/MASTER OF SCIENCE THESIS

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Utfyllende tekst/Extended text:

### **Background:**

Failure of the cement sheath is one of the most common causes of well integrity problems. The effect of failure on the well integrity is the potential for leakage of reservoir fluids and gases, and can have a large impact on the production of an oil field. Poor cement-formation bonding is a typical cause of such failure. The quality of the cement-formation bonding is dependent upon several factors, such as the type of formation, type of cement, and type of drilling fluid used during drilling. This Master project is based on experimental work. The student will study the cementing of various formation rocks and measure the mechanical bond strength between the cement and rocks (with and without drilling fluid between the cement and rock).

### Task:

- 1) Describe the background of the study and the experimental setup.
- 2) Determine the cement-formation rock bonding strength with mechanical tests with different formations and drilling fluids.
- 3) Discuss the findings and the implication towards well integrity.

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# Preface

This master thesis report is done under the course "TPG-4920" at the Department of Petroleum Engineering and Applied Geophysics, Norwegian University of Science and Technology, Trondheim, Norway. The experimental work was carried out at Formation Physics lab of SINTEF petroleum research.

The objective of this thesis work is to give an insight to the students in their field of specialization. Working on a project individually, gives more understanding and enhance skills to manage the task in given frame of time. Working in the lab and performing experiments gives the idea of the working environment in practical field. This thesis work is compulsory for the completion of the master's degree program at NTNU.

# Acknowledgement

All praise belongs to Almighty Allah Who created the world.

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My heart is filled with gratitude for my supervisor, Sigbjørn Sangesland, Professor at the Department of Petroleum Engineering and Applied Geophysics, NTNU whose inspiration, motivation and stimulating suggestions enabled me to complete this research work and report.

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In the end, I express great tribute to my parents for being the source of love, never ending support and having confidence in me. I would also like to thank all my friends who always stood by me. The completion of this thesis work was not possible without the help and guidance of all of them.

Waqas Mushtaq

# Summary

This experimental work is the continuation of the literature study of cement-formation bonding, done in the autumn semester. Cement-formation bonding has not been given special attention so far, in resolving the well integrity problems. A lot of work has been done on other influential factors but this area has not been studied much. With a view to have better understanding about this unexplored area, the experimental setup was developed and several experiments were carried out. Although some experiments were done previously on this topic but that work was limited to two specific rock types.

Four different rock types, including sandstone, limestone, chalk and shale were used in experiments. The results obtained from these experiments are discussed in this report. The rock samples were also treated with the drilling fluid to observe the effect of drilling fluids on the bonding strength at cement-formation interface. These results were, then compared to the dry samples of the same rock type and difference between bonding strength was measured.

This research work will be helpful in understanding the failure occurred at the cementformation interface. By knowing all these facts affecting the bond strength, it is possible to improve the bonding strength by treating them carefully. Future work in this area can be valuable to overcome the well integrity issues. This page is left blank intentionally.

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### **1. Introduction**

Well integrity has been an important aspect for all E & P companies in managing the reservoirs during the whole life of the well. Protecting the environment, working in safe conditions and maximizing the productivity from wells are some key factors for companies. The life span of well and productivity is of no use if it poses a threat to the environment or human life. A total of 74,000 wells were drilled worldwide during 2001. A major concern in majority of these was the sustained casing pressure (1). This sustained casing pressure is usually caused by poor cementing job that provides insufficient zonal isolation. Achievements have been made to attain improved well integrity and complete zonal isolation with the evolution of technology. Improved cementing techniques, material selection and new design approaches have been implemented to ensure the safety and zonal isolation.

Cementing is considered as most important and vital factor to serve as well barrier. Several well barriers are placed to achieve well integrity and to make whole operation safe. A well barrier is an envelope that prevents the unnecessary flow of hydrocarbons into the well or into other productive zones (2). Both primary and secondary well barriers are important and placed in such a way that if one fails then other will be active. NORSOK D-010 is being used in Norway as a standard for operations. Cementing comes in primary well barriers so it makes it more important to do successful primary cementing job.

The cement is used for the well construction to seal the formations, zonal isolation and to stabilize the well. All this is accomplished to prevent the flow of hydrocarbon fluids into the well bore or into other producing zones. The quality of the cement ensures the safety and life span of the well productivity. The confirmation of good cementing job can be done by using several tools including temperature logging, radioactive tracers and acoustic logging (3). Several kinds of cements are in use for well construction depending upon the nature, properties of formation and depth. All these kinds are presented in appendix A1.

The foremost step in achieving long term zonal isolation is to have successful primary cementing job. It is very important that the pumped cement get a complete hydraulic bond with both, casing and formation. The failure in the primary cementing job may lead to a lot of risk and failure to zonal isolation and well integrity. There are several factors that influence the primary cementing job. These include improper removal of drilling mud, formation of

filter cake, formation behavior, shrinkage of slurry and lost circulation. All these factors change the properties of either cement slurry or rock formation and make it incapable to form a hydraulic seal at interface.

Drilling mud alters the properties of the formation and can contaminate the cement slurry as well. The drilling mud must be removed properly before the cementing job. The filtrate from the drilling mud penetrates into the formation, leaving the small solid particles at formation interface. This led to the formation of filtercake at formation face. This filtercake does not allow cement slurry to form a strong bond at interface. So, it becomes the weakest point for failure to occur. In case of high pressure, fluids especially gas can migrate easily along this interface which can cause an increase in the annulus pressure. In case of the oil based mud, the formation interface becomes more critical for strong bonding and then it needs to be treated properly to remove filtercake as much as possible (4).

Shrinkage of cement slurry is another serious issue in failure of the primary cementing job. It can be caused by the lost circulation zones. Another property of the cement is the loss of volume due to the curing. When cement slurry is cured the volume decreases due to the fact that the reactants have larger volumes than the products in the chemical reaction. Cement cannot be placed to the desired height which creates a weak point for the undesired flow of formation fluids. Filtrate from the cement slurry also penetrates into the formation, thus reducing the required volume. Slurry setting time or thickening time is also very important. If it sets before reaching the desired height, complete isolation cannot be attained.

The behavior of the rock formations have always been complex. A better understanding of formation behavior and properties can be helpful to attain improved well integrity. The rocks behave according the minerals present in them. These minerals come into contact with placed cement differently, while the composition of the cement slurry is kept same throughout the selected interval. So, as a result bonding at the interface could be less than optimal, thus complete zonal isolation is not obtained.

In order to resolve well integrity issues particularly at cement-formation interface, remarkable work has been done by Carter and Evans (5). They used Indiana limestone and Berea sandstone in their experiments. They studied shear bond strength, compressive and hydraulic bond strength by treating the rock samples with different drilling fluids and cementing them against different cement slurries.

The findings of their experiments related to cement-formation interaction are as follows:

- The maximum bond strength was observed in dry cores.
- Zonal isolation cannot be achieved without removing the drilling mud from the borehole.
- Presence of filtercake on cores minimizes the strength of hydraulic bond.
- Effective bond strength was obtained when effective mud removal techniques were used.
- The intimate contact of slurry with the formation determines the strength of hydraulic bond.

These findings were further carried out by H.K. J. Ladva, who studied the bond strength at shale-cement interface (6). He studied all the factors involved in achieving the strong bond at cement-formation interface. He performed the experiments with dry cores and also by treating them with OBM and WBM. Using only shale formation, he performed adhesion test, shale swelling test and cement-formation bond strength with and without filtercake. He concluded his results as follows:

- Bond strength was found maximum in case of dry shale samples.
- Treatment of shale samples with OBM and WBM reduces the bond strength at interface.
- When the cement was placed against a mud filtercake, the failure plane was within the mudcake and gas flows through this path.
- Water transport in case of shales, also affects the bond strength.

This thesis work is a part and contribution to the work that has been done before. This work is different from the previous ones in a way that it includes the study of shear bond strength for the rock types including sandstone, limestone, shale and chalk. With an idea to study the shear bond strength at cement-formation interface for all the rock types, an experimental setup was developed. Tests were made on dry samples and fluid treated samples as well. Two kinds of WBM and two kinds of OBM were used in these experiments. A comparison of difference in the bond strengths is also presented.

# 2. Literature Review-Cement Formation Bonding

The objective if this chapter is to present an overview of the research work done so far to resolve the well integrity problems. Since, there are various factors that come in well integrity but in this report, the focus is to address only those factors which are important and influential for cement-formation bonding.

Cementing the casing for zonal isolation is one of the most important factors for efficient well integrity. Cement is accepted as a permanent WBE together with casing and cement inside the casing or borehole. During drilling, coring, tripping and completion operations, it is normally taken as secondary WBE, after drilling mud and BOP. Placement of cement, type of cement, cement additives and thickening time are some essential factors affecting the sealing ability of cement sheath. The longevity of a well depends on a good cementing job. The main functions of cementing include:

- Zone segregation and isolation.
- Casing support and preventing it from corrosion.
- Formation stability.
- Fluid movement restriction between permeable zones.
- Close an abandoned portion of well.

### 2.1 Types of Cement

The main component in all kinds of drilling cements is Portland cement. It is made by burning a blend of limestone and clay (7). The slurry consists of Portland cement, water and different additives and then placed into the bore hole. Slurry properties are adjusted according to the borehole conditions by adding retarders and other additives. These compounds change the physical properties of the cement which include density, thickening time, filtration rate and viscosity.

American Petroleum Institute (API) classified cements into 9 categories from A to H and J. These categories are defined according to the material used, depth, formation type and pressure temperature conditions. These cements are also graded as ordinary (O), moderate sulfate resistant (MSR) and high sulfate resistant (HSR) (8). The types of cement and their use according to depth are presented in appendix A1. These classes are limited to a certain depth and formation condition requirements.

#### **2.2 Cement Placement Techniques**

Two main types of cement placement techniques are being used in practice. Cement is placed according to the required applications. Cementing a liner is quite different from the casing because casing extends to the surface while liner is attached to the bottom of the last cemented casing. Squeeze cement job is performed in lost circulation zones or leak cemented zones to stop the undesired communication. Both techniques are well illustrated in figure 2-1. Figure 2-1(a) is showing cement placement for steel casing while figure 2-1(b) is illustrating cement placement in liner string. The liner is cemented where the last casing has already been cemented.



(o) CEMENTING CASING STRINGS (b) CEMENTING LINER STRINGS

Figure 2-1: Illustration of cement placement in casing and liner string (7).

#### 2.2.1 Primary Cementing

The primary cementing refers to the conventional single stage cementing of casing strings and liners. This operation is performed after the casing has been run in the hole. This is accomplished by pumping cement slurry down inside casing and displaced it out into the annular space between the casing and the borehole. The cement is then allowed to set before drilling is resumed or the well is completed. The major objective of primary cementing has always been to provide zonal isolation in oil, gas, and water wells that is to exclude fluids such as water or gas in one zone from oil in another zone in the well (9). To achieve this objective, a hydraulic seal must be created between the casing and cement and between the cement and the formations, while at the same time preventing fluid channels in the cement sheath.

#### 2.2.1.1 Mechanism of Primary Cementing

Figure 2-2 is showing the typical primary cement placement technique. The mechanism of primary cementing involves the usage of bottom plug and top plug. In the first phase of mud

circulation, drilling mud is circulated out of the hole. This is done by the use of displacing fluids and sometimes with spacers. The spacers and chemical washes also help to clean the borehole and keep the cement slurry separate from drilling mud. It is presented in second phase of figure 2-2. This helps in prevention of contamination by the interaction of slurry and drilling mud. Sometimes scratchers are used to clean the hole and reduce the thickness of mud cake. More often, a mud preflush is used to minimize these undesired effects.



Figure 2-2: Illustration of different stages for typical primary cementing including circulation of mud, spacers and displacement of cement slurry (9).

In the third phase, cement slurry is displaced in the borehole followed by the bottom plug. When the desired volume is pumped into the string, a top plug is released from the plug container. The displacement fluid is pumped to push the top plug. The difference between these both plugs is that the top plug has a solid rubber while bottom plug has a thin rupture diaphragm. When further pressure is applied, the diaphragm of the bottom plug ruptures. Then the slurry is pumped into the annulus between casing and formation. When top plug reaches the bottom plug, increased pressure shows the completion of cementing job.

The well is kept shut for some time to set the cement slurry. During this time, pressure inside the casing should be maintained carefully. Sudden decrease in pressure may cause leaks and fracture in settled cements. The advancement in the technology has introduced many new methods but primary cementing technique is still predominant and preferred (7).

#### 2.2.2 Secondary Cementing

Secondary cementing comes in many different names and with many procedures. Remedial cementing, squeeze cementing, stage cementing, multiple string cementing, reverse circulation cementing and plug cementing, all comes in it. The purpose of secondary cementing includes:

- Reduce the hydrostatic pressure on weak formations to prevent fracture.
- Reduce the pumping pressure of the cement pumping equipment.
- Cementing selective formations.
- To repair leaks from previously cemented formations.
- Mostly cement is not required in widely spaced intervals.

#### 2.2.3 Multistage Cementing

Multistage cementing is performed when there is failure in job and cementing could not provide complete zonal isolation. It is used to repair the leak zones which have already cemented, stop the movement of gas migration and to do some remedial jobs. The stage cementing can be two stage separately, continues two stage and three stage. The most commonly used is two stage separate cementing while the latter two are very complex and rarely used (8). Any small single failure in these jobs may lead to the failure of whole operation. So, they are used in very specific conditions.

#### 2.2.4 Squeeze Cementing

Squeeze cementing is a process used to attain complete zonal isolation of annulus. In this technique a relatively high pressure is applied to force the cement into weak zones behind the casing. It is also used to isolate a production zone by sealing off adjacent nonproductive zone

(7). Casing repairs such as joint leaks or corroded casings are also functions of squeezed cementing.

In high pressure squeeze operations, high hydraulic pressure is applied to make new channels by fracturing the rock. Then pressure forces the slurry to enter in these new channels. Low pressure squeeze operations are used where the permeability of the structure or formation is sufficient enough to move slurry in it. Hesitation method can be applied in both operations to have good results. Infect this methods appears to be more effective than continuous pressure application. In this method, pressure is applied after small intervals.

#### 2.2.5 Liner Cementing

A liner is a string that does not extend to the surface, but it is anchored or suspended at the bottom of last set casing. The difference from the casing cementing is that the bottom plug is not used in this process which results in the contamination of slurry. In this case, dual liner wiper system is preferred. The setting tool and drillpipe are pulled out at the completion of job before cement gets hardened.

#### 2.3 Important Factors for Efficient Mud Removal

The quality of the bond depends mainly upon the efficient removal of drilling mud, fluid loss, formation of filtercake, displacement techniques, well geometry and lost circulation (1). Drilling mud alters the properties of the formation and stick to the exposed formation which leads to the formation of filtercake. This weak point can be the cause of flow of hydrocarbon fluids into the wellbore or into other productive zones. The cement slurry is designed according to the formation type and behavior but it does not remain much effective when it comes in contact with the altered formation. So, efficient removal of mud can provide the basis for strong bonding of formation with cement. Some important factors are discussed here for efficient mud removal from the borehole and to improve the well integrity.

#### 2.3.1 Fluids Flow Pattern

Fluids flow pattern is important during the fluid movement in the borehole. Two types of flow patterns are observed. They are laminar flow and turbulent flow. In turbulent flow, particles move in swirled motion and the velocity along the walls is almost the same as in the center of borehole. While in laminar flow, the velocity at the walls is low due to high friction

and the maximum velocity is at the center. The figure 2-3 is showing the laminar and turbulent flow patterns of fluids.



Figure 2-3: Illustration of fluid flow patterns (10).

Field experiments show that improved turbulent flow should be preferred for proper removal of drilling mud (11). The preflushes and cement slurry should also be placed in turbulent flow if possible. But turbulent flow may cause high flow rate, high friction pressure and risk of losses in narrow sized holes (12). So, in these conditions, effective laminar flow is recommended. Pump flow rate, mud rheology and densities are changed to achieve a relatively flat interface.

#### 2.3.2 Density Differential

As a general rule, density of displacing fluid is always kept higher than the fluid being displaced. The figure 2-4 is showing an illustration what can happen in case if this rule is violated. In figure 2-4,  $\rho$ 1 is displaced fluid density while  $\rho$ 2 is displacing fluid density. In case of figure 2-4 at left side, mud cannot be removed effectively due to low density of displacing fluid. While the figure at right side shows the stable interface due to high density of displacing fluid.



Figure 2-4: Effect of light displacing fluid. It causes improper removal of mud (left). Stable interface is achieved by having dense displacing fluid, results in efficient mud removal (right) (12).

#### 2.3.3 Friction Pressure Gradient

Friction pressure of displacing and displaced fluids is important in a way to make better sweep efficiency. Fingering mechanism can be developed in case of improper friction pressure. For better results, it is suggested that friction of displacing fluid must be greater than displaced fluid (12). This will result in flat and stable interface between fluids. The figure 2-5 is explaining the same mechanism. Left hand side figure is showing fingering effect as a result of low friction pressure of displacing fluid.



Figure 2-5: Fingering phenomenon caused by using low viscous displacing fluid (left). No fingering effect by using higher viscous displacing fluid (right) (12).

#### **2.4 Effects of Filtercake**

The formation of filtercakes on wellbore walls is influential on bonding strength. Mudcake properties, thickness and behavior should be understood completely before the selection of cement slurry (13). Filtercakes are formed across the permeable sections as the filtrate from the mud penetrates into the formation leaving the solid particles at borehole walls. The failure of filtercake also causes some problems. Filtercake failure can be in two ways. The cake detaches in large slabs from the rock surface which is called liftoff mode. The other failure could be in a way that it can create pinholes by developing small erosion channels through which hydrocarbons can flow (14). If the cake ruptures and there is no proper clean up, then well may need some other interventions.

Various techniques have been developed to understand the characteristics of the filtercake. Among them, scraping technique is considered efficient which can measure the strength and moisture profiles of typical filter cakes with a 0.1 mm resolution (14). The better understanding of the behavior, strength and properties could be helpful to remove filtercake efficiently before slurry placement. Fluid loss is the penetration of the mud filtrate into the formation as a result of high pressure gradient. Fluid loss occurs during drilling when drilling mud is being circulated in the borehole. The same phenomenon occurs when the filtrate from cement slurry penetrate into the formation when it is placed against the formation. The aqueous part of the slurry penetrates into the porous formation and results in increasing viscosity of the slurry. Some of the solids may penetrate into more porous formation and form an internal mud cake.

This filtercake can be valuable in a way that it can help in strengthen the cement-formation bond (15). Increased number of solid particles can be helpful in strong bonding between cement and formation. Increased viscosity can displace drilling fluid effectively. But this benefit becomes ineffective as it alters the rock properties and results in reduced production and weak bonding strength (16). Mud cake thickness is dependent on drilling fluid composition and hydraulic conditions. The typical thickness of mud cake could be 2-5 mm (6). Dehydration of slurry increases compressive strength, low permeability and shrinkage. Slurry viscosity can cause a high friction pressure during placement. It may shorten or increase the thickening time and total volume of the slurry could by decreased by dehydration. To achieve effective isolation, fluid loss control is very crucial. Field experiences and observations suggest to maintain the amount of fluid loss (9). Fluid loss should be of a certain limit that it could change properties according to our requirements of borehole conditions. Hartog et al. defined a general maximum fluid loss rate of 200 mL/30 min for oil wells and 50 mL.30 min for gas wells (17).

Filed measurements show that it is hard to measure the direct effect of fluid loss in primary cementing. However, this effect can be measured better in remedial cementing. Change in pressure can give an idea of fluid loss measurement but slurry compressibility should be known. To avoid the risks and gas migration, filter cake properties should be understood completely. Fluid loss additives can be added into slurry. But this may increase the cost of the slurry. So, economic factors and borehole conditions are decisive to include fluid loss materials.

#### **2.5 Lost Circulation**

Lost circulation is another severe problem which mostly happens during drilling and sometimes during cement placement. Cementing in weak formations may lead to the lost circulation due to low fracture gradient (18). The drilling fluid or cement slurry starts penetrating into formation voids and fractures. As a result fluid does not reaches up to the annulus and volume is reduced. It also damages the rock formation and reduces the pressure on the formation and may cause another formation to flow into the bore hole.

During cementing, lost circulation reduces annular coverage, causes poor zonal isolation and reduces safety. Most often, this lost circulation problem is solved before primary cementing. But if it happens again during cementing, there are numerous ways and techniques to stop it. Stage cementing or remedial cementing can be done to avoid this problem. The right decision at right time can eliminate this problem and ensures complete zonal isolation.

#### **2.6 Formation Behavior**

For effective isolation, cement should set against the unaltered formation. The presence of mud cake and behavior of shale formations limit the cement to isolate rock formation completely. Formation mineralogy also affects the cement-formation bonding.  $Fe^{+2}$ ,  $Ca^{+2}$ ,  $Mg^{+2}$  and many other minerals are present in the rocks and they act with the fluids according their properties. Their effects can be minimized if the chemical composition of the drilling fluids, spacers and cement slurry are set according to them. Soluble evaporite minerals can also challenge the bond integrity(9).

#### **2.7 Causes of Poor Cementing**

Cementing is considered as the most critical part of the well completion. Several factors qualify the cement job during drilling for hydrocarbons. The poor cementing occurs due to following factors.

#### 2.7.1 Insufficient Cement Coverage

Inadequate removal of drilling mud is the main cause of the failure in cementing. Pockets of the drilling mud and filter cake prevents the cement to form a strong bond with the formation (19). To remove them properly and clean the borehole before cementing, spacer or pre-flushes are used. Their use help in displacing drilling mud from the borehole and also reduce the filtercake effect.

#### 2.7.2 Gas Migration

Gas migration is possible the three different ways. One is between casing and cement interface. Other is at cement formation interface due to the presence of filter cake on formation face. Third is due to the cracks developed during cement setting phase due to pressure fluctuations (20). All these three types of gas migration can be stopped by successful primary cementing job.

#### 2.7.3 Insufficient Mud Height

Cement is lost in high permeable or highly fractured zones. This loss results the reduction in slurry volume. So slurry cannot achieve required height and a lot of portion is left uncemented (4). This is known as cement fallback in the annulus. The exposed and uncemented area then becomes a weak point for the flow of hydrocarbons.

#### 2.8 Use of Oil based Mud

The use of the synthetic based mud makes it more difficult to remove the filter cake from the formation.OBM has been proven to leave a layer on the formation while displacing cement slurry. This oil layer prevents the slurry to form a strong bond with the formation. This results in the poor bonding at cement-formation interface. Spacers and chemical washes are needed to for a long time as compared to the WBM (4). The spacer rheology, stability and compatibility become very important in the case of OBM. Experiments show that 100 % oil based mud filtrate causes the reduction in permeability (21).

#### 2.9 Experimental Studies of Sedimentary Rocks

Carter and Evans performed a series of experiments using Indiana limestone and Berea sandstone cores to find the bonding strength between cement and formation. Unfortunately, their findings for sandstone left incomplete as failure occurred within the core rather than at bonded interface. The dry cores were made in contact with cement slurries. In one case cement was squeezed while in other it was not squeezed against the formation. Afterwards, soft and tough mud cakes were developed on cores and then they were made in contact with slurries (5). Cores were also tested by treating them with drilling mud and then their interaction with slurry was observed.

Figure 2-6 is representing hydraulic bond strength, shear bond strength and compressive strength for the dry limestone. Seven different kinds of cements were placed against the dry lime stone. According to it, bonding strengths are maximum in case of using API class A cement and Latex cement.



Figure 2-6: Bonding strength of dry core by cementing with different kinds of slurries (9).

In next case, cores were treated with water based drilling mud first. Then the cement was squeezed against the cores at a pressure of 100 psi. The mud cake was removed in this case. A significant decrease in shear bond strength can be observed in figure 2-7 as compared to the dry cores.



Figure 2-7: Bonding strength of a core after treatment with water based drilling mud and then placed against different slurries after the removal of mud cake (9).

In figure 2-8, the cores were treated with water based drilling mud. In this case cement was not squeezed against the cores. Mud cake was also removed before placement of slurry. It can be observed that there is a significant impact of squeezing the slurry. The bond strengths were more in the previous case where cement was squeezed.



Figure 2-8: Bonding strengths of core against the placement of different muds after the removal of mud cake. Slurry was not squeezed in this case (5).

Figure 2-9 is showing the bonding strengths of cores when cement was squeezed and the walls of the cores were not cleaned.



Figure 2-9: Bonding strength of cores with cements squeezed in the presence of mud cake (9).

In the next case, cement was not squeezed against the cores with mud cakes present on the walls. Figure 2-10 is illustrating the bonding strengths of the cores in this particular case. The hydraulic bond strengths and shear bond strengths were low as expected but the compressive strength was almost same. The formation of mudcake hinders the strong bonding within formation and cements.



#### Experimental Study of Cement-Formation Bonding

Figure 2-10: Bonding strength of cores with cements not squeezed in presence of mud cake (9).

The findings of Evans and Carter were a big step in this particular subject of casingformation interface. Further remarkable work was done by H. K. J. Ladva who presented it in more descriptive way by performing some experimental work with shales. He suggested that good understanding of clay minerals can be beneficial for the selection of slurries.

#### 2.10 Experimental Work on Shale Formation

A comprehensive work was done by H. K. J. Ladva on shale formations. He conducted experiments in laboratory and studied the behavior of shale formation and slurry in different cases. He worked on Catoosa and Oxford shales and treated them against class G cement. This report contains an overview and findings of his experiments.

The potential for shales to swell when exposed to cement slurry was observed. Unconfined linear swelling tests were performed with two shales. Smectite readily swells in the presence of water. From figure 2-11, it can be observed that after exposure to a synthetic cement filtrate with the specific composition, the Oxford shale swelled more than the Catoosa shale.



Figure 2-11: Swelling of shale cores in synthetic cement filtrate (6).

#### 2.10.1 Bond Strength Measurement

A cell was designed to measure the shear bond strength between the shale core, filtercake and set cement. A freshly cored shale plug with a diameter of 1 in. [25.4 mm] and a nominal length of 0.8 in. [20 mm] was placed centrally on the acrylic plastic base s shown in figure 2-12. An acrylic plastic spacer was then placed on top of the shale core, and the cement slurry was poured around shale to the top of the spacer.



Figure 2-12: Shear bond testing cell with shale formation (6).

After the curing period, the base, lid, and acrylic plastic spacer were removed. The cell walls and cement ring were supported while a brass rod of 0.8 inch in diameter was used to load the shale core.



Figure 2-13: The push out profile of Oxford shale from cement (9).

The maximum force required to push out the shale at a rate of 0.5 mm/min was measured. The shear-bond strength was calculated by dividing the maximum force by the area of the shale-cement interface. A typical shear test profile, presented in Figure 2-13, shows the maximum load that the interface could withstand before rupturing. During the shear-bond test, the untreated Oxford shale core was pushed out of the cement ring, but a film of shale

remained on the inside of the cement. This suggests that the shear-bond strength of the shalecement interface was greater than the shear strength of the shale itself.

#### 2.10.2 Effect of Filtercake

The effect of filtercake on bonding strength was also observed. The cores were treated with WBM, OBM, and washed WBM. The results are presented in figure 2-14.



Figure 2-14: Shear bond strength with and without filtercake (6).

It can be seen from figure 2-14 that the bonding strength is high in case of dry core. The shear bond strength for the OBM is weaker than the WBM. The reason is that the OBM does not interact with cement. A small improvement was observed in case of WBM when it was followed by a chemical wash.

This section has summarized what is known and has done practically about the cementformation interface. The quality of the bond at this interface is affected by many variables, including the formation composition, drilling-fluid composition, cement-slurry composition, temperature, and pressure.

### **3. Experimental Setup**

The objective of this thesis work was to establish a setup to study and analyze the shear bond strength at cement-formation interface after setting of cement slurry. The shear bond strength at cement-formation interface can be defined as the resistance of rock movement against the placed cement. This resistance determines the strength of the shear bond at cement-formation interface. This preliminary study will be helpful to form a strong bond at cement-formation interface and to overcome well integrity issues. By keeping all the previous literature and experimental work in consideration, a setup was established to identify problematic issues.

#### **3.1 Materials and Equipment Used**

The materials and equipment used in all experiments are as follows.

#### 3.1.1 Cement

Class G cement is used in these experiments to cement around the rock samples. The compositions and properties are presented in appendix A2.

#### 3.1.2 Rock Types

Four rock types were selected to measure the bond strength at cement formation interface. These are sandstone (Castlegate), limestone (Piatra Lecesse), chalk (Lixhe) and shale (Mancos). The shale sample was kept in oil for long term storage. All other three rock types were dry initially but later they were treated with other fluids for experiments. The shale samples were kept in oil before the sample preparation. The rock samples were cut in to cylindrical shape pieces with specific dimensions. These dimensions were kept almost constant to measure the bonding effect on same sized samples. Rock samples were cut into cylindrical shape with specific size of 13.5mm in diameter and 15mm in height. The dimensions are presented in the appendix A3, which also contains the bond strength calculations.

#### 3.1.3 Drilling Fluids

Four types of drilling fluids were used to immerse the rock samples. These were obtained from the field and then prepared in the lab by adding some additives as suggested by the provider. Both water based and oil based drilling fluids were used. Water based fluids include FormPro and Glydril while oil based include Versatec and Warp. These fluids are written as WBM1, WBM2, OBM1 and OBM2 respectively. The drilling fluids were prepared by shearing for 10 minutes with a rooter-stator at 6000 rpm. Then the bridging particles were added and mixed with propeller at 500 rpm for 10 minutes.

#### 3.1.4 Steel Chamber

The rock samples were cemented in a steel chamber, manufactured in workshop. The chamber consists of two half cylindrical shaped segments. These segments were joined together by screws. The dimensions are 26mm in diameter and 30mm in height. Several chambers were manufactured to prepare more samples at same time. The steel chambers were designed in a way to place the rock sample in the center and then set the cement around it. A piece of hard plastic with the same diameter was placed on the rock sample. The following figure 3-1 is showing the schematic of the sample preparation. Friction free spray was used on the walls in order to take out the sample smoothly.



Figure 3-1: Schematic diagram of sample preparation in steel chamber.

#### 3.1.5 Pressure Chamber & Heating Oven

A pressure chamber was used to cure the cemented rock samples. The pressure was kept constant at 15 bar. It also helped in keeping the shrinkage factor at minimum. The prepared samples were then placed into the pressure chamber. This pressure chamber then placed into a heating oven to cure the samples. The temperature was kept constant at 66  $^{\circ}$ C.

#### 3.1.6 Push-out Tester

A push-out tester was used to push the cemented rock sample out of the placed cement around it. The diameter of the pushing rod was slightly less than the diameter of rock sample. The aim was to push the rock sample at the interface between rock sample and placed cement. The movement speed was kept constant before and after the breakage point. The parameters were specified before performing the tests, which are presented in appendix A4.

#### **3.2 Methods**

Five samples were prepared from each rock type, one dry while four treated with the drilling fluids before cementing.

#### 3.2.1 Application of Drilling Fluid

The rock samples were immersed in selected drilling fluid. The rock was allowed to soak for 30 minutes, taken out and then kept at normal temperature and pressure for 10 minutes to flow off excessive drilling fluid. These rock samples then kept in the steel chamber and cement was placed around them.

#### 3.2.2 Cement Properties and Mixing

The cement was prepared by mixing the dry cement and water with a water/cement mass ratio of 0.44. The cement was mixed with a handheld mixer for almost 25 minutes. The mixing procedure was kept according to the equation 3.1, which shows that the mixing procedure should equal an energy input of approximately energy in kJ/kg.

$$E/m = k\omega^2 t/V \tag{3.1}$$

Where k is a constant (1.6  $10^{-11}$  m<sup>5</sup>/s),  $\omega$  is the rotation speed (rad/sec), t is the time (sec) and V is the volume (m<sup>3</sup>) of the mixed slurry.

#### 3.2.3 Placement of Cement

. The mixed cement slurry was then poured in the steel chamber until the cement height equals the half of the hard plastic piece length.

#### **3.3 Characterization**

The cured samples were taken out from oven and pressure chamber after 7 days. These samples were then taken out of the steel chambers and hard plastic pieces were removed by pushing out gently. Sometimes samples need to be grinded to make the surface smooth from top and bottom before the push-out test. Cemented samples were again placed in the steel chambers to perform the push-out test. A force was applied to push the rock sample to move

in the cement ring. This force at which the sample started moving in the ring was measured. The sample was moved almost 4mm to 6mm for different samples, depending upon the cracks and breakage of the cement ring around them. By combining the force and the area between rock and cement, shear strength was calculated as the measure of bonding strength. The friction force between rock sample and cement is neglected in the calculations.

### 4. Results and Discussion

The results from all these experiments are described below individually according to the rock type. The measurement of the shear bond strength is presented in graphical form for each rock type.

#### 4.1 Comparison of Dry Rock Samples

In the figure 4-1, a comparison of dry rock samples for each rock type is shown. This shows the bonding strength of each rock type with the cement. The shear bond strength was not calculated along the interface between formation and cement except the shale sample. In shale sample, the bond strength is exactly at the cement-shale interface. But in case of all other rock samples, the actual failure occurred in the rock sample itself. This value indicate that the bond strength at the cement-formation interface is higher than the rock compressive strength. The same results were also observed in experimental work of H.K.J. Ladva for shale and Evans and Carter's work for sandstone and limestone.



Figure 4-1: Graphical representation of shear bond strength for dry rock samples.

As seen in the figure 4-1, the highest bond strength was obtained in the case of limestone sample. But it is not the actual bond strength as failure occurred in limestone sample. The reason can be the highest compressive strength of limestone as compared to other formation types. The failure of the limestone sample is shown in figure 4-2. A thin layer of limestone sample is visible inside the cement ring. The cracks in the cement ring also affected the measurement of bond strength. The limestone sample moved relatively easy after the cracks in the cement ring.



Figure 4-2: Limestone sample after push-out test showing cracks in the cement and thin layer of limestone inside the cement ring. This shows the failure within limestone sample instead of cement-formation interface.

From figure 4-1, minimum bond strength was observed in the case of dry sample of chalk. The behavior was similar to the limestone sample as it is not representing the actual bond strength at the interface. The application of the force resulted in the movement of the rock sample but leaving a thin layer of chalk inside the cement ring. This minimum bond strength is actually due to the low compressive strength of the chalk sample. Figure 4-3 is showing the layer of chalk inside the cement ring. The measurement of the strength was affected by the creation of cracks in the cement ring.



Figure 4-3: Dry sample of chalk after push-out test. A couple of cracks and a thin layer of chalk are visible inside the cement ring, showing failure within the chalk sample.

Sandstone sample also behaved in the same way. A thin layer of sandstone can be seen inside the cement ring in figure 4-4. Several cracks were developed in case of sandstone sample.



Figure 4-4: Dry sandstone sample leaving the thin layer inside the cement ring. Several cracks developed as a result of push-out test.

The actual bond strength at the cement-formation interface was measured in the case of shale sample. The movement of the shale sample was exactly at the interface between shale and cement. The figure 4-5 is showing the moved shale sample within the cement ring. There is no layer of the shale on the inner side of cement ring which confirms the actual bond strength measurement. This bond strength at the interface was also influenced by the cracks in the cement ring. This means that the shear bond strength at the interface was less than the compressive strength of shale sample.



No shale layer inside the -cement ring. It shows the actual measurement of bond strength exactly at the interface.

Figure 4-5: Dry shale sample after push-out test. The shale sample move exactly at shale-cement interface, while some cracks were developed during shale sample movement.

#### 4.2 Limestone

The results from the limestone samples are represented in this section. In the previous section 4.1, it was observed that the limestone has the strongest bond among all other types of rock samples. So, it is important to see the behavior of limestone samples after treatment with different drilling fluids. The effect of the drilling fluids on bond strength is illustrated in figure 4-6. This figure is representing the raw data obtained from the push-out test. The purpose to present figure 4-6, is to show the fluctuations in the force to move the rock sample. Ideally, when the rock sample starts moving once, then the force required to move the sample further should be less than the breakage point. But in some cases it was observed more than the breakage point. The reasons for this behavior could be increased friction at the cement-rock interface as the sample moves down. This friction is also dependent on the grain sizes of the rock samples.



Figure 4-6: Graphical representation of data obtained from push-out test. It shows the amount of the force required is increasing to move the rock sample within the cement ring alongwith distance.

The figure 4-7 is representing the shear bond strength for limestone sample with and without drilling fluids.



Figure 4-7: Graphical representation of the shear bond strength for limestone samples with and without drilling fluid.

A significant reduction in bond strength can be seen after the treatment with drilling fluids. Treatment with water based fluids reduced the bond strength considerably as compared to the dry sample as shown if figure 4-7 above. The movement of the rock sample was exactly at the interface between cement and formation. The figure 4-8 is showing the limestone samples after the push-out tests.





In case of the oil based mud, a huge reduction in bond strength can be seen from figures 4-6 and 4-7. The presence of thin layer of oil has a large impact on bonding. A very thin layer of oil can be seen at the interface in the figure 4-9. Immersion in oil made the rock sample weak

for bonding with cement. This bond strength is minimum bond strength in all cases for all rock types.



Figure 4-9: limestone sample treated with OBM 2. Reduction in bond strength is due to the thin film present at the interface.

This behavior can be explained as a function of rock properties i.e. porosity and permeability. If the limestone is porous and permeable, then immersion of limestone sample in OBM resulted the penetration of oil constituents from OBM. This caused the reduction of porosity and permeability of the sample. The oil layer prevented the rock to form a strong bond with cement.

#### 4.3 Sandstone

The behavior of sandstone samples is illustrated in figure 4-10. The samples were treated with WBM 1 & 2 and OBM 1& 2. The maximum bond strength was fond in case of dry sample as discussed above in section 4.1. Treatment of sandstone samples with WBM resulted in the significant reduction of bond strength. The bond strength in case of WBM 1 is almost the same as that of limestone treated with WBM 1.



Figure 4-10: Graphical representation of the bond strength of sandstone sample with and without drilling fluids.

But it increased in case of sandstone when treated with the WBM 2 as compared to limestone sample. There is not much difference in bond strength when sandstone sample were treated with the OBM 1 &2.

#### 4.4 Shale

The behavior of the shale samples is illustrated in figure 4-11. In general, the bonding strength in shale samples is higher from all other rock types when treated with drilling fluids. However, this is not true in case of dry samples.





The WBM did not affect the bonding strength of the shale samples as they did in case of other rock types. The reason for this behavior could be low porosity and permeability of shale samples. Shale samples were also kept in the oil before getting into contact with the cement. It is possible that samples were saturated with the oil already so they could not absorb more

water during immersion in WBM. The bond strength is also maximum in case of OBM 2 as compared to the all other rock types.

During the push-out tests, all shale sample move along their interface between shale and cement. Although, few cracks were developed but still the failure did not occur in the rock itself. This behavior also clarifies that the bond at the interface was not stronger than the rock's compressive strength even in the case of dry sample.

#### 4.5 Chalk

The results from the chalk samples are presented in this section as no one has studied it before. So, it is important to show the behavior of the chalk samples after treatment with the drilling fluids. The behavior of the chalk samples is presented in graphical from in figure 4-12.





After treatment with the water based mud, the bond strength at the interface was low as compared to the dry sample. The presence of the drilling fluid on rock sample reduced its ability to form a strong bond with the cement. The movement of the rock sample was exactly at the interface between cement and formation. The figure 4-13 illustrates the movement of chalk sample along its interface after the push-out test.

#### Experimental Study of Cement-Formation Bonding



Figure 4-13: Chalk sample treated with WBM 1 after push-out test. The sample moved along the interface.

Bonding strength is case of oil based mud is much lower than the dry and water based fluids treated samples. This could be due to the non-sticking property of oil constituents in mud. Oil saturated rock samples lose their ability to stick strongly with the cement. The movement of the rock sample was exactly at the cement-formation boundary. But a thin layer of the oil can be observed in figure 4-14.



Figure 4-14: Chalk sample treated with OBM 2. Presence of thin layer of oil constituent reduces its tendency to form strong bond with cement.

#### 4.6 Possible Explanation for Increase in Force after Failure

The graphs in figure 4-15 are representing the raw data obtained from push-out test. The objective to present this raw data is to give possible explanation for the increase in required force for the movement of rock sample even after failure. The behavior of the shale sample was different from all other rock types. It is almost the same presented in section 2.10.1 in H.K.J. Ladva's work. Ideally, the required force after failure should be less than the force at the point of failure.



Figure 4-15: Raw data for all rock types from push-out test.

There could be several reasons for this unusual behavior. This increase can be explained as:

#### 4.6.1 Swelling of Samples

One possible reason could be the swelling of the rock samples during the immersion in drilling fluids. Swelling of rocks depends on the mineralogical compositions of rock. Rocks containing only anhydrite do not swell, whereas, rocks containing 5 percent clay may develop a swelling stress (22). It is possible that during the immersion of rock samples in the fluids,

the samples were swelled and increased their diameter slightly. The hard plastic spacer kept on the sample was almost equal in diameter initially. So, now after soaking rock samples, the hard plastic piece diameter reduced equally to the swelling of sample. So, the increase in force could be caused by this cement at reduced diameter at hard plastic's end.



Figure 4-16: Illustration of swelling of rock sample during immersion in drilling fluids. This increased diameter caused the increase in force for the movement even after failure. (a) Plastic spacer and rock sample before immersion in drilling fluids. (b) Increased diameter of rock sample after soaking in drilling fluids, which is now more than plastic spacer. (c) Increased diameter caused more force to move rock sample further down because of placed cement just beneath the bottom edges of the rock sample.

The figure 4-16 is illustrating the phenomenon of increased diameter of rock samples during soaking in drilling fluids. In figure 'a' the diameter of plastic spacer and rock is same before soaking in drilling fluid. In figure 'b' the diameter of the rock sample has increased slightly than the plastic spacer. While figure 'c' is representing the condition just before the push-out test and after the removal of plastic spacer. It can be observed that the placed cement has been set equally in amount as the swelled rock sample in the lower part. So, this cement could be the cause of resistance and increase in force required to move the sample after failure.

#### 4.6.2 Smoothness of Samples

The samples were cut with the same coring bit. But it is possible that the smoothness of the outer surface of samples would not be the same. It is dependent on the grain size of each rock type. So the cavities at the surface may cause the cement to move in. As a result the bonding at the cavities would be more than the smooth plane.

#### 4.6.3 Radial Stress

Another possible reason could be radial stress in the sample during push-out test. This can also be written as effect of Poisson's ratio on the rock properties (23). The applied force can cause the rock sample to stretch radially. So when it stretches, more force is needed to move it further. When the radial stress increased from a certain limit, it produced cracks in the cement, which were produced in many samples and can be observed in figures above.





Figure 4-17 is showing the radial stress that caused the increment in force to move the rock sample further.

#### **4.7 Different Shale Behavior**

The shale samples behave differently from all other rock types. From figure 4-15, it can be seen that shale samples behaved ideally after the failure during push-out test. This can be explained as the shale samples were oil saturated before cementing. So these samples were not swelled while soaking in drilling fluids. The diameter of the shale samples remained same before and after the immersion in drilling fluids.

#### 4.7.1 Bond Strength of Shale Samples Treated with WBM

Bond strength of the shale samples was found to be high as compared to other three rock types in case of treatment with WBM. These shale samples were kept in the oil before cementing and curing. So, it turned out to be an interesting observation of strong bond strength of oil saturated samples with water based treated fluids.



Figure 4-18: Shear bond strength of rock samples treated with WBM1.

Ideally shale should not have this much strong bonding while treated with WBM. The oil saturated rock does not make strong bond with cement. But figure 4-18 is showing the opposite behavior. This could only be explained as a function of rock and fluid properties. The presence and distribution of the minerals can make it more receptive for WBM. We do not know the exact composition and properties of all the minerals present in shale rock sample. We also do not know the exact composition and properties of the drilling fluids used in experiments. So, this unusual behavior cannot be explained anymore before knowing the composition and properties of both, shale sample and drilling fluids.



#### 4.8 Effect of Filtercake on Shear Bond Strength

Figure 4-19: Illustration of shear bond strength of all rock types.

Figure 4-19 is showing the shear bond strength for all the rock types treated with different drilling fluids. This figure is summarizing the effect of filtercake or drilling films for all the rock samples used in this experimental work. A significant reduction in shear bond strength can be observed clearly.

The formation of filter cake, in case of OBM has made the rock worse for strong bonding with cement, while shear bond strength is comparatively high in case of water based mud. The behavior of the shale samples is quite interesting in above figure. The shale samples have strong bonding strength in all cases except in case of OBM1. The possible reasons for this behavior have already explained in section 4.6 above. This behavior highlights the significance of effective removal of mud from borehole and filtercake from the formation face.

# **5.** Conclusion

On the basis of the results from experiments, following conclusions are drawn:

- Ideally maximum bond strength can only be obtained against the unaltered rock formation.
- Rock properties such as porosity, permeability and saturation are also important factors to attain good bond strength at interface.
- Use of water based mud reduces the bond strength at interface between cement and formation. But this effect was found minimum in case of shale samples. This effect was either due to immersion of samples in oil or low porosity and permeability.
- The use of oil based mud reduces the bond strength at interface between formation and cement. The presence of a layer of oil constituents on formation prevents it to form a strong bond with cement.
- Zonal isolation and complete well integrity is associated with effective removal of drilling mud from the borehole before cementing.
- The value of shear bond strength did not change much in case of shale samples as compared to other rock types, even after using different drilling fluids.
- The oil saturated rocks can have strong bonding with cement slurry, depending upon the mineral constituents present in that rock.

# 6. Future Recommendations

A better understanding of the influential factors on cement-formation interface can improve zonal isolation and resolve well integrity problems. The results of this preliminary experimental work can be used as a basis for future work on this particular issue. For complete zonal isolation, more comprehensive work can be done by keeping these considerations in mind.

- The data obtained in these experiments is not 100% relevant to the field cases. Actual field data can be taken and studied in future.
- In petroleum well it is likely that the cement is being placed against filter cakes and not just drilling fluid films. So, future work should include the measurement of shear bond strength with filtercake and not with drilling fluid film.
- Different kinds of cements should be used against the same formation to observe whether the change of cement has an effect on shear bond strength or not.
- During the push-out tests, several cracks were developed which affect the exact measurement of the shear bond strength at interface. It is also important to measure the cement ring compressive strength and use it to find the exact shear bond strength at interface.
- Friction force between rock sample and cement is not included in measuring the shear bond strength. "This phenomenon could be reduced with a more sophisticated sample cell, where the cement sheath is placed under a controlled compression. This friction factor can be included to obtain the exact value of the shear bond strength at interface.
- The mineral composition in a rock sample is very important. It would be help to know the exact mineral composition of the rock samples to study the unusual behavior of rock sample, as shale behaved in these experiments by having a strong bonding with water treated samples.
- The rock properties such as porosity, permeability and saturation are the function for strong bonding. So, their consideration can give a better understanding.

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# 8. Abbreviations

- API American Petroleum Institute
- **BOP** Blowout Preventer
- **CEC** Cation Exchange Capacity
- HSR High Sulfate Resistant
- LCM Lost circulation material
- MSR Moderate Sulfate Resistant
- MTA Mud to Agglomerated Mud Cake
- MTC Mud to Cement
- **OBM** Oil-base mud
- **OPC** Ordinary Portland cement
- SWBS Small wellbore simulator
- WBE Well Barrier Element
- WBM Water-base mud
- **YP** Yield point

# 9. List of Symbols

Symbol	Description
Ε	Energy, kJ
m	Mass, Kg
t	Time, sec
V	Volume, m <sup>3</sup>
ω	Rotation Speed, rad/sec
ρ	Density, Kg/m <sup>3</sup>

# **10. Appendices**

# Appendix A1

Туре	Grade	Approx. Depth	Requirements
		(II)	
			When special properties are
Class A	O, MSR to HSR	Surface to 6000 ft	not required
			Moderate to high sulfate
Class B	MSR to HSR	Surface to 6000 ft	resistant material conditions
			required
Class C	MSR to HSR	Surface to 6000 ft	When conditions require high
			early strength
Class D	MSR to HSR	From 6000 ft to	For high temperature and
		10000 ft	pressure conditions
Class E	MSR to HSR	From 10000 ft to For high temperature	
		14000 ft	pressure conditions
		From 10000 ft to	For extremely high
Class F	MSR to HSR	16000 ft	temperature and pressure
			conditions
			Covers a large variety of
Class G	MSR to HSR	Surface to 8000 ft	depths by addition of
			accelerators and retarders
			Covers a large variety by
Class H	MSR to HSR	Surface to 8000 ft	addition of accelerators and
			retarders
		From 1200 ft to	Covers a large variety by
Class J	MSR to HSR	16000 ft	addition of accelerators and
			retarders

Table 1: Types and grades of cement according to their requirements and depth

### Appendix A2

# **Class G Cement- Properties and Composition**



# TEST CERTIFICATE NORWELL, API Class G, Well Cement

-

TESTED IN ACCORDANCE WITH API SPEC 10A / NS-EN ISO 10426-1

Address: Setrevelen 2 P.O.Box 38 N-3991 Brevik	Phone: +47 35 57 20 00 Telefax. +47 35 57 04 00	Ent.no: NO 934 949 Bank accou 6003 06 124	9 145 VAT Lilleakerveien 2b nt. P.O.Box 143 Lilleaker 488 0216 Octo
NORCEM A.S	0.00 %	0.75 % Max.	
Na <sub>2</sub> O Eq	0.60 %	0.75 %	cases and y men ager
2*(3 CaO*Al <sub>2</sub> O <sub>3</sub> )	17.5 %	24.0 % Max	Laboratory Math ager
4 CaO*Al <sub>2</sub> O <sub>3</sub> *Fe <sub>2</sub> O <sub>3</sub> +		0.0 /0 midA.	Vill HIL W
3 CaO*Al <sub>2</sub> O <sub>3</sub>	1.3 %	30% Max	Colorer to 2012
		48.0 % Min	Brevik October 18 <sup>th</sup> 2012
3 CaO*SiO <sub>2</sub>	64.7 %	65.0 % Max.	
Insoluble Residue	0.08 %	0.75 % Max.	
L.O.I.	0.62 %	3.0 % Max.	
SO3	2.00 %	3.0 % Max,	
MgO	1.72 %	6.0 % Max.	TIME IN HOURS
Cr 6+	0.00 mg/kg		
CHEMICAL COMPOSITIO	<u>: NC</u>		
Thickening Time (100 Bc)	100 Minutes	90-120 Minutes	ø m
Minute Stirring Per. :	14 Bc	30 Bc Max.	AU 11 11 11 11 11 11 11 11 11 11 11 11 11
Schedule No. 5:			CONSIST
THICKENING TIME TES		1000 par Min.	EN COX 80
60°C, Atmos, Pressure :	2451 psi	1500 psi Min	80
38°C, Atmos. Pressure :	890 ps)	300 psi Min	
COMPRESSIVE STRENG 8-Hour Curing Time:	<u>GTH :</u>		100
Free Fluid	4.3 %	5.9 % Max.	
Fineness, Blaine	320 m²/kg		Schd, 5 Schd. 7g
PHYSICAL TESTS :	Results :	Requirements :	
Delivery No.: FD20-12			2298 tons Cement Class G, shipped from our works by mbc Conberria bound for Agotnes, Mongstad and Dusavik October 15 <sup>th</sup> 2012.



# NORWELL, API Class G, Well Cement

#### ADDITIONAL TESTS

DELIVERY No.: FD20-12

#### **SLURRY DENSITY :**

44 percent Water By Weight of Cement

16.00 lb/gal

#### RHEOLOGICAL PROPERTIES :

Viscometer Dial Reading at 300 rpm	82
Viscometer Dial Reading at 200 rpm	70
Viscometer Dial Reading at 100 rpm	54
Viscometer Dial Reading at 60 rpm	46
Viscometer Dial Reading at 20 rpm	35
Viscometer Dial Reading at 6 rpm	20
Viscometer Dial Reading at 3 rpm	15
Plastic Viscosity (PV) = 1,5*(A-B)	42 cP
Yield Point (A-PV)	40 lbf/100 ft2
10-Minute Gel Strength, 3 rpm, max.	17 lbf/100 ft2
<ul> <li>After 30 sec. stirring</li> </ul>	15 lbf/100 ft2
<ul> <li>After 1 min. stirring</li> </ul>	16 lbf/100 ft2
<ul> <li>After 2 min. stirring</li> </ul>	16 lbf/100 ft2

#### RETARDER RESPONSE :

Schedule No. 7g 185°F with 0,4% R6-AB

Max. Consistency 15-30 Minutes Stirring Period

7 Bc

156 Minutes

Thickening Time, 100 Bc

kcs.

Brevik, October 18th 2012

fur 2 1206

Laboratory Manager

NORCEM A.S

Address: Setrevelen 2 P.O.Box 38 N-3991 Brevik Phone: +47 35 57 20 00 Telefax: +47 35 57 04 00 Ent.no: NO 934 949 145 VAT Bank account; 6003 06 12488 Head Office: Lilleakervelen 2b P.O.Box 143 Lilleaker 0216 Oslo

# Appendix A3

Rock type	DF	Rock sample diameter	Length	Area	Force	Pressu	ire
		d (mm)	h (mm)	A (m2)	(N)	Ра	Psi
	WBM1	13,49	15,54	0,000659	899	1365041	198
	WBM2	13,46	15,45	0,000653	1417	2168929	314
Sandstone	OBM1	13,50	15,70	0,000666	435	653288	95
	OBM2	13,45	15,78	0,000667	363	544410	79
	DRY	13,45	15,75	0,000666	2747	4127670	599
	WBM1	13,55	15,41	0,000656	887	1351696	196
	WBM2	13,60	15,73	0,000672	927	1379308	200
Limestone	OBM1	13,62	15,94	0,000682	293	429587	62
	OBM2	13,60	15,31	0,000654	111	169538	25
	DRY	13,54	15,53	0,000661	3119	4721060	685
	WBM1	13,54	15,00	0,000638	463	725685	105
	WBM2	13,54	14,93	0,000635	580	913269	132
Chalk	OBM1	13,54	14,97	0,000637	280	439710	64
	OBM2	13,42	15,04	0,000634	244	384172	56
	DRY	13,54	14,97	0,000637	1882	2955482	429
	WBM1	13,60	15,47	0,000661	1924	2910884	422
	WBM2	13,59	15,53	0,000663	1401	2113437	306
Shale	OBM1	13,60	15,47	0,000661	363	549195	80
	OBM2	13,58	15,54	0,000663	865	1304244	189
	DRY	13,59	15,50	0,000662	2418	3653668	530

Table 2: Dimensions and strength calculation for each rock type

# Appendix A4

### Table 3: Parameters for push-out test

Break sensitivity	15 %
Data acquisition rate	0.5 Hz
Data acquisition rate fast	2.0 Hz
Maximum load	999.0 N
Pre-load	5.0 N
Pre-load speed	0.5 mm/min
Test speed	0.5 mm/min
Test speed 2	0.5 mm/min