POLITECNICO DI TORINO

Department of Environment, Land and Infrastructure Engineering

Master of Science in Petroleum Engineering

Evaluating alternative cementing material for remedial operations

Thesis Advisor:

Prof. Raffaele Romagnoli, Politecnico di Torino

Prof. Dr. Kris Ravi, Montan Universitat Leoben

Thesis Author:

Viktoriya Rostoshanskaya

Dedication

I would like to dedicate this master thesis to my dear Farther who sadly passed away far too early.

Thank you for always being here for me, helping me and supporting me in every single minute of my life. You will forever remain in my heart. May your soul rest in peace…

Acknowledgement

First, I would like to express my sincere gratitude to everybody in the Drilling and Completion Engineering Department of Montanuniversität Leoben. Especially Mr. Michael Prohaska, who allowed me to participate Mobility Program in Austria and be part of Well Integrity Platform project. Thank to my advisor professor Kris Ravi for clear project guidance and professional recommendation during my work. It has been a great learning experience being part of a DPE Team that works together for deliver benefits into industry. Thanks to Mr. Alexander Fine for all discussions we have had throughout project. Thanks to Mr. Asad Elmgerbi for his constructive comments regarding drilling and cementing operation process, who has enhanced this thesis.

I would also like to thank Professor Raffaele Romagnoli for his acceptance of being my advisor from Politecnico di Torino, and his valuable suggestions throughout both working on my project report and master thesis. Thanks for answering all the emails I have sent and for the interesting technical discussions we have had over the last few years.

In addition, I am thankful to WellSet company especially Mr. Usher for supporting with required equipment for MRBC system during experimentation stage.

Finally, I would like to thank all my fellow students and professors at Politecnico di Torino for making these Master program an incredible journey in Italy.

Abstract

Conventional Cement used for oil well-cementing operation worldwide mainly because of availability and economical reason. Along with their significant advantages there are many jobs where cement sheath could fail and thus compromise the integrity of the well. The problem with conventional cement is, that in some situation it may not be reliable to provide zonal isolation, prevent migration of fluid, set successfully without shrinkage and set quickly. Remedial operations to repair cement failures leads to additional personnel and material inputs and rig time expenses. The evaluation of the most promising technical failure mechanism focusing thereby on compressive strength development despite contamination and the possible identification of additives that can improve compressive strength and anti-shrinkage properties of the conventional slurry may increase the cement performance. The verification of this most crucial parameter was based on failure analysis done through literature review and proof of concept (POC) laboratory simulation.

The object of this thesis is the development of comparison experimental study which includes the 4 stages of laboratory testing with different slurry composition that can shows compressive strength development pattern and sedimentation-shrinkage properties. Overall objective is to study, evaluate, and define alternative cementing materials used for remedial jobs.

The thesis covers a detailed literature review which describes the fundamentals of well integrity during wellbore operation, characteristics of Portland cement and additives influence properties of the slurry, technical evaluation of conventional cement failure and briefly description about alternatives to cement. This will be followed by an experimental part includes (POC). The final step will include suggestion for application in oil field with conclusion and further work recommendation with plans for more specific experimental research.

List of Figures

List of Table

TABLE OF CONTENT

Chapter 1 **Introduction**

In 21st century innovative efforts made to improve the monitoring of bottom hole parameters during the life cycle of the well that could assist the industry to detect and prevent well integrity issue, avoid barrier elements failures and reduce unwanted repair cost, increase production recovery and safety as well as improve the profit of assets. Energy industry's demonstrated ability to innovate and continually improve well-construction technology that may be more valuable to all plays if effective construction and workover techniques are provided will highly focused on HSE and more integrated approach in well protection by implying double barrier envelope failure-free system while drilling and production. In other words, to ensure safety those two independent barrier envelopes are required in all types of well operations. Drilling operation divided them into a primary and a secondary barrier element. By killing severe and complete loss circulation zones subsequently preventing a kick is the first line of defense to reduce the probability of getting a blowout. The unwanted influx of formation fluid into the wellbore comes from losing the well control, the primary barrier element, thus creating a kick. If this kick is not detected or handled properly, it may result in a blowout if the secondary barrier element or more precisely blowout preventer fails or is not initiated. The same tike wellhead body with annulus access ports and valves, seals and casing hangers with seal assemblies and surface tree can be attributed to secondary barrier elements. Preventive and risk-reducing measures are important to mitigate and be able to control these undesired events. Knowledge of primary well barrier elements like cap-rock, cement sheath, casing, production packer, completion string, downhole safety valve combined with their behavior during drilling operations became an important topic over last decade. The Importance of having the barrier elements in place and the ability to understand them and to test and maintain during the life cycle of the well should not be underestimated. This approach is described by a primary barrier element in direct contact isolation material with the formation and a secondary barrier envelope outside of this, as a hat-over-hat arrangement. This is a robust solution to safeguard well activities, and no matter where the pressure tries to escape, there will be protection behind it. This will also allow the repair of an element while the other envelope is containing the pressure. Envelope of two barrier philosophy will help contain the pressure and hydrocarbons and prevent the incident from escalating into an accident. Different requirements are imposed to provide an effective work of both barriers. (ISO/TS 16530-2, 2014)[1,8]
The nature of those procedures related to observing and verifying the integrity of the primary and secondary barrier envelopes as referred to in standards, ISO 16530-1, ISO 16530-2 and NORSOK D-10 Rev4 latest one developed by Norwegian Petroleum Industry. Other manuals and best practice from operator companies are widely used alongside mentioned in previous sentence.

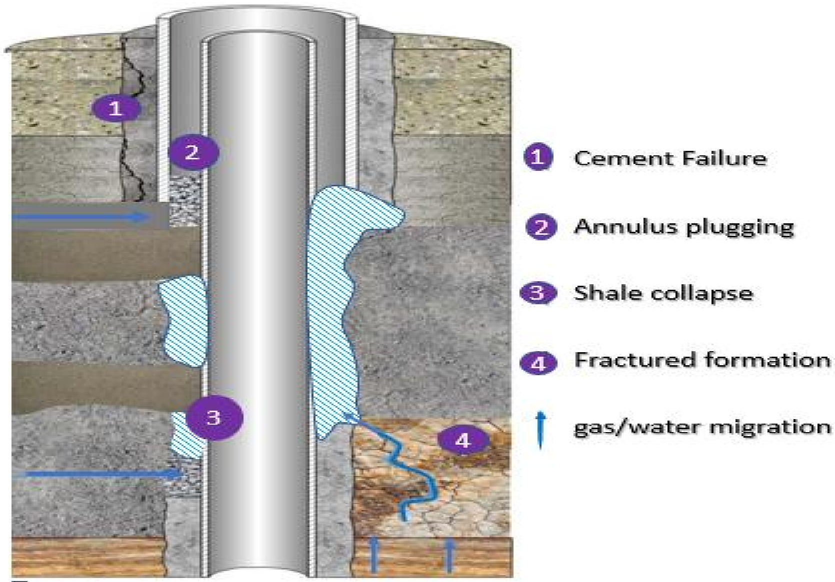


Figure 1: Fluid migration paths (Schlumberger, 2014)[2]

Figure 1 represented fluid migration due to several problems that can occur during or after cementing operation. Number 1 shows fluid migration that has occurred due to cement sheath failure and number 2,3 indicate how well integrity issue may occur due to casing failure and shale collapse. Number 4 shows how gas/water leak paths occurred due to shrinkage and poor bonding between cement and casing/formation. Since the concept of migration takes place it could mean that there has been a loss of zonal isolation occurred, therefore, cement sheath, plugs, packers, casings, and cap-rock all those primaries barrier elements that must provide safety during well construction and production were failed. Damage to any barrier element will not compromise the entire integrity of the well directly, but it can increase the risk in the future conditions. Therefore, failure of one barrier element could lead to a decrease in energy production, increase unwanted zone production, loss of zonal isolation, diminish the economic outlook of the well and gradually corrode/degrade other barrier elements. It is basic to comprehend the reason and reliance of each single obstruction component and the idea of the boundary envelope. (API-RP-53, 2012)[3]

Experience from industry has shown in the present investigation is that many of the well integrity issues come from the fact that despite the good mechanical properties of cement and economical relevance, it still brittle, display low tensile strength, shrunk and cracked with time. This is the major reason why enhancing performance in cementing jobs has not occurred and thereby causing permanent challenging, deterioration and the significant increase in costs of providing well integrity. New components, better chemical additives, and specifically designed alternative cementing materials are enablers to provide zonal isolation in increasingly challenging environments, including deep water, long-reach wells, wells with sour gas environment and wells subjected to large high temperature and high-pressure cycles throughout the life of the well. (PSA,2017)[4]

Overall objective of this master thesis is to study, define and evaluate alternative cementing materials that can be used for remedial jobs. To meet the overall objective the research plan should pass through the following chapters:

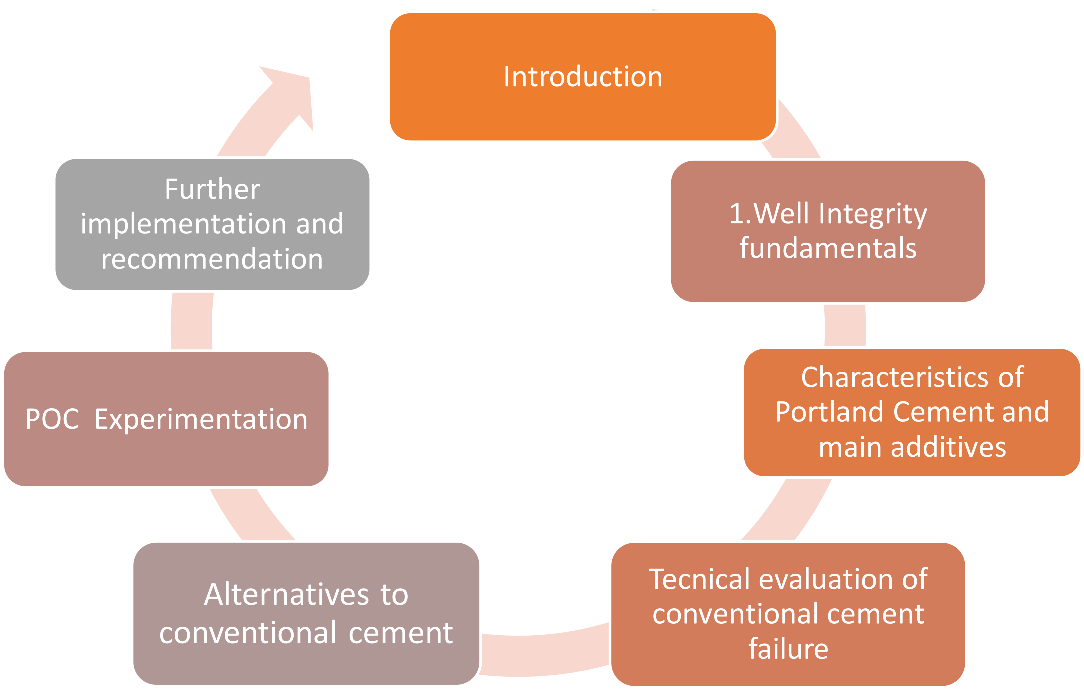


Figure 2: Structure of Master thesis project «Evaluating alternative cementing materials for remedial operations

Chapter 2 **Well integrity fundamentals**

Well integrity always has been a very important topic for the entire life of the well and this chapter describes the main aspects that will cover a huge range of the problem includes the description, hazard and accident conditions, design and function of well barrier, warrant of well integrity during the well operations. (AWT, 2010)[7,8]

2.1 Description

According to NORSOK D-10, Well integrity is defined as: "application of technical, operational and organizational solutions to reduce risk of uncontrolled release of formation fluids throughout the life cycle of a well". This functional standard sets the minimum requirements for the equipment/technics to be used in a well, but it leaves it up to the operators to pick up the solutions that meet the requirements. Following from this definition, the whole planning of drilling and completion process of wells will ought to perceive the answers that provide HSE (Health, Safety and Environment) during the well life cycle. Some other implication is that operators and service companies have a responsibility to make sure that the equipment planned to be used will comply with the standard and if no longer, the equipment will need to be improved and certified before use. Deviations from the standard must be made in some cases when the standard allows this. On the off chance that an answer chosen strays from the standard, this arrangement should be proportionate or better contrasted with necessity.

While selecting technical solutions, it is critical to set the right equipment specifications and define the requirements for the well barrier to ensure the well integrity is maintained all through the well life. Fundamental things to specify are the BOP evaluation and size, the casings for use, the pressure range on downhole and wellhead equipment and the material specification for them. These specifications will be set at an initial stage of assignment and the afterwards selection of equipment must be primarily based on it. (API-RP-53, 2012)[3]

The oil and gas industry define wells as a physical asset which join the reservoir to the surface, and via which we produce hydrocarbons and formation fluids. In order carry successful production and control the flow of fluids resident within the formations and reservoirs which the well penetrates the well must be designed carefully according to NORSOK D-10. Wells have a finite productive

life, which commences with the main drilling operation.
Further, the well may be subjected to a variety of operation steps including completion, stimulation, intervention, maintenance and the last one abandonment.
Effective control of well integrity desires to keep in mind this range of well operations in addition to regular state production. Integrity is variously defined in dictionaries as a state which guarantees authenticity, reliability, continuity and uprightness; a state which can be relied on as a correct condition at any factor within the life of a well. (NORSOK D-10)[5,6]
Summing up, NORSOK D-10 standard defined well integrity as a condition of a well in operation process that has fully functional and two qualified well barrier envelopes. Any deviation from this regulation is an insignificant or essential well integrity breakage. Common integrity problems are mainly associated with leaks in tubing or valves, however it can be also associated with lack of zonal isolation. Any aspect that leads to a functional failure is a loss of well integrity. The challenge is identified the likelihood of some future adversity scenarios.

2.2 Hazard and accident conditions

Defined hazard and accident conditions (DFUs) is responsibilities of all companies to identify the potential occurrences of major failure that needs to guard against during operation. DFUs within the petroleum industry according to The Norwegian Petroleum Safety Authority (PSA) study include the following directions:

- Leaks of flammable gas or liquids:

A distinction is drawn between ignited and non-ignited leaks. A non-ignited leak, for example, could allow gas to spread over large areas so that later ignition causes an explosion and a major accident.

- Well control incidents:

Loss of well control could lead to a blowout. Such an incident has the potential to cause substantial harm to people, the environment and material assets.

- Fire/explosion in other areas:

An example of such incidents is a fire in the living quarters with the potential to develop into a major accident.

- Collisions and other structural damage to a facility:

A distinction is drawn between collisions by vessels (supply ships, shuttle tankers or the like) maneuvering close to the facility, and by vessels not related to the activity or drifting objects (such as barges).

Platforms and rigs are designed to withstand minor collisions. But being struck by a big vessel, possibly at high speed, could cause damage which leads in the worst case to complete collapse of the support structure. (An Introduction to Well Integrity, 2012)[11]

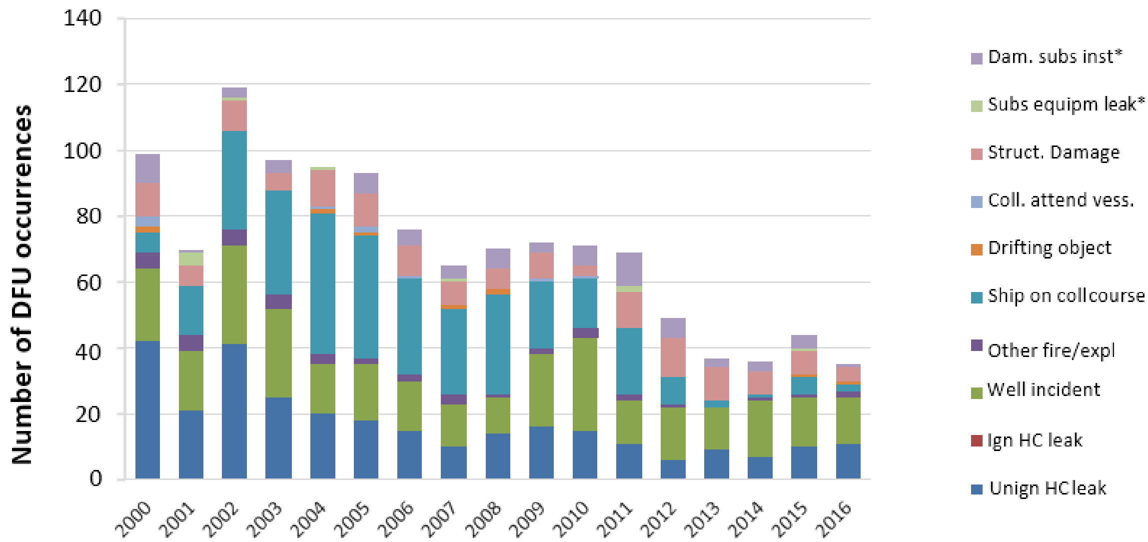
Damage can also occur in extreme weather conditions. This type of hazard involves loss of stability or mooring/positioning system failures on floating units, resulting at worst in a total loss.

- Leaks from subsea production facilities with pipelines and associated equipment:

Installations on the seabed can be damaged by objects dropped from above. Fishing gear may also cause substantial harm.

The major accident potential of damage to subsea facilities relates primarily to pollution from possible oil spills. Any nearby surface facilities could also be threatened. (PSA, 2018)[9]

Figure 3 shows the trend in the number of reported DFUs in the period 2004-2016. It is important to emphasize that this figure does not take account of the potential of near- misses in respect of loss of life. This means that a fall in the total number of incidents does not necessarily entail a fall in the total indicator. There was a rising trend in the number of incidents during the period 1996-2000, which has been discussed in previous years' reports and is therefore omitted from the figure. After an apparent peak in the number of incidents in 2002, there is a gradual reduction in the number of incidents with major accident potential. Since 2013, the number of incidents of this type has been relatively stable per year. There was a small peak in 2015, but the number of incidents in 2016 is the lowest recorded in the period (RNNP 2016)[10]



*Within the safety zone

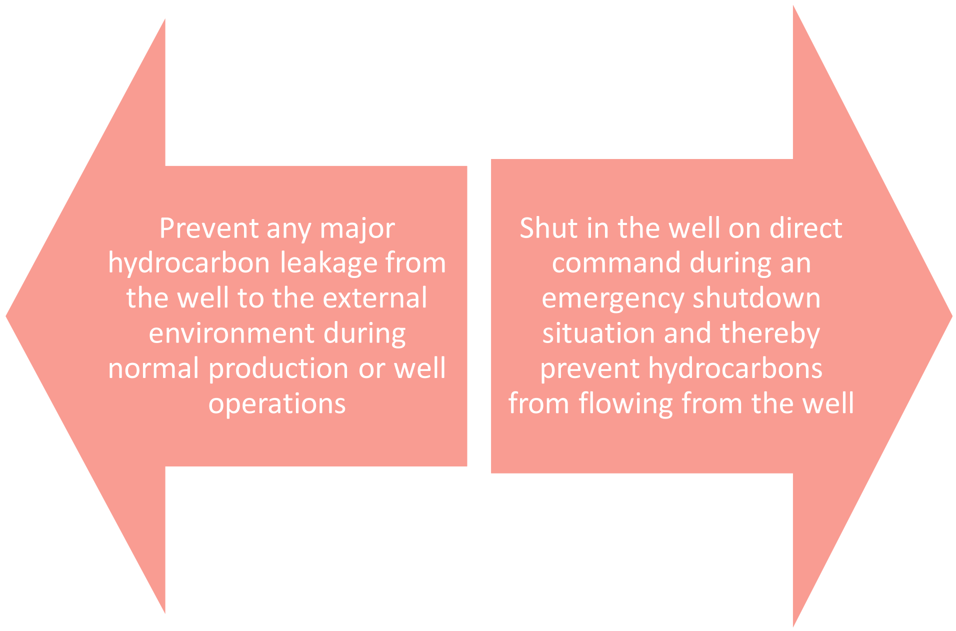
Figure 3: Reported DFUs (1-10) by categories

All possible failure must be accomplished in sufficient detail and especially for the facility, plant, location and operation, so that you can identify the situations that the individual barrier element has a position in handling. Realistic combinations of failure, hazard and accident situations ought to be assessed, since such combinations might also detect the scale of barriers.

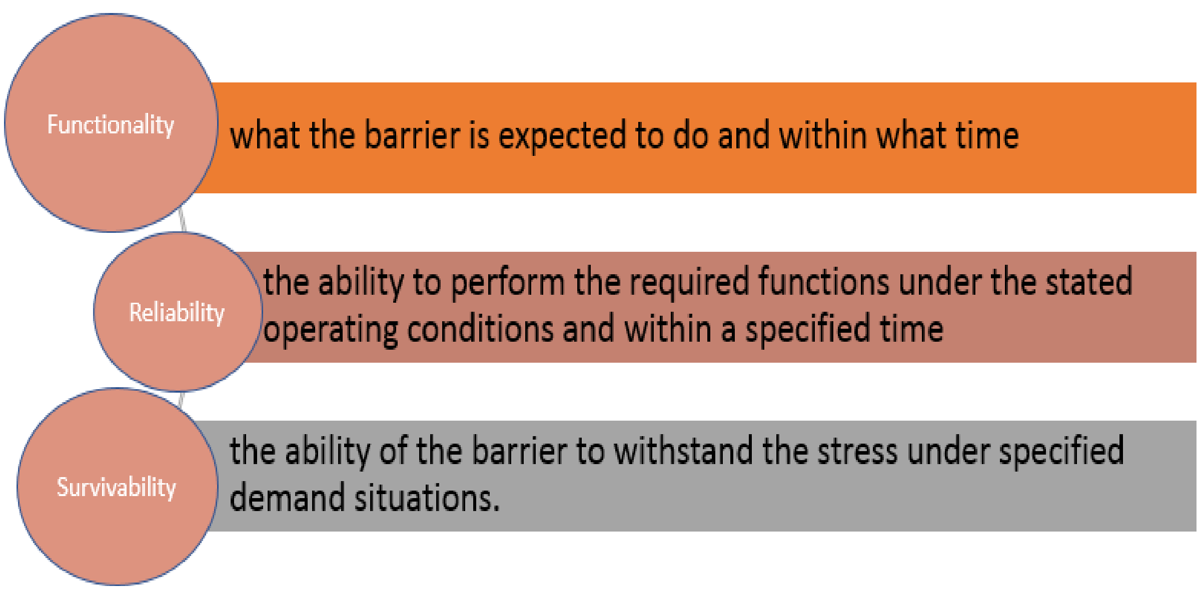
2.3Well Barriers

NORSOK D-10 specifies that: "There shall be envelope of two well barriers elements available during all well activities and operations, including suspended or abandoned wells, where a pressure differential exists that may lead to uncontrolled flow from the wellbore to the external environment". Well barrier elements are used to prevent leakages and reduce the risk associated with drilling, production and intervention activities. The envelope of one or several dependent barrier elements preventing fluids or gases from flowing unintentionally from the formation into another formation or to surface. NORSOK D-10 distinguishes between *primary* and *secondary* well barriers elements. If the primary well barrier is functioning as intended, it will be able to contain the pressurized hydrocarbons. If the primary well barrier fails (e.g., by a leakage or a valve that fails to close), the secondary barrier will prevent outflow from the well. If the secondary well barrier fails, there may, or may not, be a *tertiary* barrier available that can stop the flow of hydrocarbons.(NORSOK D-10)[5,6,40]

2.3.1 The main objectives of a well barrier



2.3.2 The Requirements of a well barrier



2.3.3 The Elements of a well barrier

Barrier elements contain electrical, electronic, and/or programmable electronic technology referred to as safety-instrumented functionality. Safety-instrumented functionality are processed by a safety-instrumented system with 3 main subsystems:

Input elements: sensors (for automatic activation) or push-buttons (for manual activation)

Logic solvers: an electronic or non-electronic device that process the signals from the input elements and send signals to the relevant final elements

Final elements: physical items that interact with the well, for example valves, such that loss of containment is stopped or avoided

Safety-instrumented functionality may be built up into the same safety-instrumented system. Although, there are some important design characteristic: Functionality that correspond to the same event as kick well and choke collapse that should not share components. The primary and secondary barriers have safety-instrumented functionality, they need to be placed in two independent safety-instrumented systems to avoid a failure of the logic solver causes simultaneous failure of the primary and the secondary barrier elements. Several safety-instrumented systems can appear during installation process on oil and gas called by their essential function listed below:

- emergency shutdown systems
- process shutdown systems
- fire and gas detection systems (An Introduction to Well Integrity, 2012)[11]

2.4 Well integrity during wellbore operations

During the life of the well mainly 4 operational stages can be defined as exploration/development, construction, production and abandonment. All those stages are crucial for the well integrity. The initiation phase so called Design Stage involves exploration activities before actual wellbore operations begin. This phase is part of thesis interest comprises with drilling, cementing and completion operation during the well construction phase the stresses around the bore hole, and hence the resultant wellbore stability, will constantly change due to fluctuating gravity of the fluids inside the wellbore. These processes will influence the resultant stresses in the cement sheath and defines as focus of this study. The later operational stage the intended operational regime of the well and planned/unplanned human interventions includes changing fluids, pressure testing, perforation, production, injection, hydraulic and acid fracturing and sidetracking operation that will seriously affect the integrity of the sealing material. (Dickson Udofia Etetim,2013)[15]

Finally, abandonment phase involves plugging and abandoning wells at the end of production which have reached its economic life.

Ideally, during the drafting of the Well Functional Specifications, the extremes in well operation should be defined. Next, the impact, not only on casing or tubing design, but also on the integrity of the well sealing material should be evaluated. (Duijm and Markert, 2009)[12]

2.4.1 During drilling

Drilling activity starts with spudding and concludes with preparation of the well for testing, completion or suspension/abandonment. The drilling activity could impact wellbore stability, casing integrity and the cement integrity. Key Performance Indicator (KPI's) for the well construction stage are dominated by time factors which are often misaligned with life cycle well integrity. The one constant visual indicator success is the, time depth curve. Rarely are other performance measurements, such as quality, or design verification, given daily or 'by- phase prominence in communication of progress during construction. During well construction, the actual subsurface environment is seldom what is typically expected and planned for. Prediction, and actual deficits, in pore pressure, rock strength, reservoir fluids, rock properties, formation tops and lithology types, all contribute to load boundary shifts in the "as built" from those assumed at the design stage. Often, this load boundary change is accepted without verification of the original design due to time based KPI's. The primary purpose of the well is to ensure the safe and reliable production of fluids to surface, under the range of boundary conditions anticipated for the life cycle. During construction, controls should exist at each phase, pre and post cementation, and the well completion stage, which verifies the original design,

is still valid.
During drilling, the primary pressure barrier is the fluid column. The drilling fluid forms a filter cake to protect borehole from collapsing. This filter cake keeps the walls unimpaired that the cement would be properly placed during completion. Filter cake damages can lead to difficulties during cementing operation. (Dickson Udofia Etetim,2013)[15]
Drilling through complicated geological formations requires reinforcement of the well to avoid liquid invasion to the surface or surrounding zones. The cement column well barrier must bond to the well casing and the surrounding formation and maintain structural integrity throughout well exploration, production, and after abandonment in order to prevent remedial operation. The survey, conducted by the PSA, summed up that operator must be more focus on well integrity, especially on barrier philosophy to avoid major incidents leads to well-control issues." (Perricone, 2012)[14]

2.4.2 During abandonment

Abandonment phase is often the hardest to control operationally and because of huge challenges in industry this thesis mainly will be focus on this area of investigation. Well possessions have passed through many different cycles to get to this point. With ripeness an amount of information has been created. Often the information is not stored in a centralized easily accessible location. There are often data deterioration created by poor quality data recording, incompatible software legacy issues, and poor handover during any levels of work, which can lead to a large impact on risk management in deciding how a well can be abandoned, safely, and environmentally effectively. The majority in the uncertainties for well abandonment is often the pressure data. (Dickson Udofia Etetim, 2013)[15] When a production well exhibits sustained casing pressures, diagnosis is difficult, and restoration of annulus barriers is difficult. Often sustained casing pressures in production are accepted and managed through a dispensation process. Robustness of the dispensation and risk assessment process is variable however and some important annulus information such as hydrostatic pressure of annular fluid, and the maximum pore pressure of formation below the shoe, may not be available. It is a fact that those responsible for well integrity management in the production phase have a challenge to persuade management to install an effective well integrity management system and often even to run diagnostic tools. While one can argue that avoidance of production interruptions, limits the use of diagnostics,

risk reduction makes their use at all stages essential during the process of abandonment. (Dickson,2013)[15]
Overall, the trends define four major ways in which hydrocarbons can leak from the system to the environment are depicted on Fig.4 and the reason why that happened will comprise in the following chapter

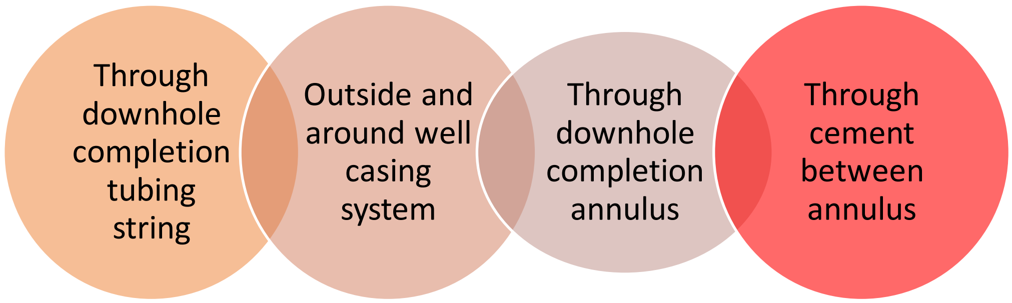


Figure 4: Ways of losing well integrity

Chapter 3 **Portland Cement and main Additives**

The main ingredients in almost all cementing jobs is Portland Cement and water as a mixing fluid to provide performance that are needed. The physical and chemical properties of well cements that changes significantly by using different compounds and additives to cement slurry that must be followed to design cement systems which will provide adequate casing protection and zonal isolation throughout the life of wells are presented in Chapter 3.

3.1 **Portland Cement description**

Portland cement is by far the most important binding material in terms of quantity produced indeed, it is possible that it may be the most ubiquitous manufactured material. Portland cement is used in nearly all well cementing operations. The conditions to which Portland cements are exposed in a well differ significantly from those encountered at ambient conditions during construction operations: as a result, special Portland cements are manufactured for use cements as well.

The chemical formulas of many cement compounds can be expressed as a sum of oxides, for example, tricalcium silicate Ca_3SiO_5, can be written as $3CaO*SiO_2$. For the simplification of the formula the abbreviations are given for the oxides most frequently encountered mentioned below

C= CaO	$F=Fe_2O_3$	$N=Na_2O$	$P=P_2O_5$
A=Al2O3	M=MgO	$K=K_2O$	F=FeO
S=SiO2	$H=H_2O$	$L=Li_2O$	$T=TiO_2$

Portland cement consists principally of four compounds: tricalcium silicate C_3S (55%-65%), dicalcium silicate C_2S (15%-25%), tricalcium aluminate C_3A (3%-8%) and tetra calcium aluminoferrite C_4AF (8%-12%). These compounds are formed in a kiln by a series of reactions at temperatures as high as 1500 degC between lime, silica, alumina and iron oxide. In the manufacturing process selected raw materials are ground to a fine powder and proportioned in such a way that the resulting mixture has a desired chemical composition. After blending, the raw material mixture is fed into a kiln and converted to cement clinker. Clinker is cooling down and a small amount of gypsum up to 5% is added, after system can be pulverized. The pulverized end product so called as a Portland cement. The compounds present in Portland cement are anhydrous. When brought into contact with water, they are decomposed forming hydrated compounds.

Supersaturated and unstable solutions are formed, gradually depositing their excess solids. Since the solubilities of the original anhydrous compounds are much higher than those of the hydration products, complete hydration should ultimately occur.

The silicate phases in Portland cement are the most abundant, often comprising more than 80% of the total material. C3S is the principal constituent, with a concentration as high as 70%. The quantity of CS normally does not exceed 20%. Majority C_3S component that contributes to all stages of strength development and one that responsible for early strength development. When C_2S hydrates slowly and affects long term compressive strength development. The hydration of C3S is an exothermic process; therefore, the hydration rate can be followed by conduction calorimetry. From the thermogram given in Fig.5 the five hydration stages were defined.

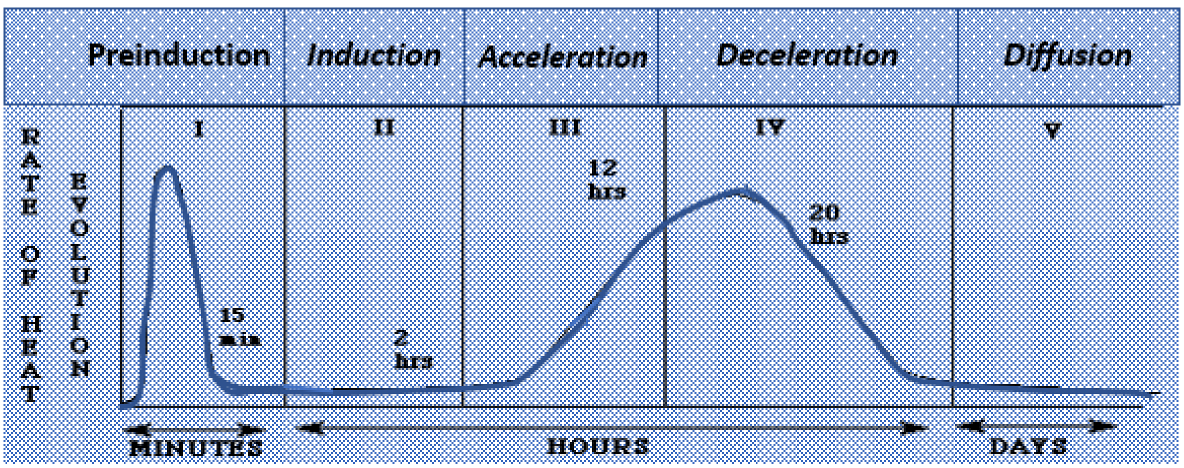


Figure 5: Hydration stages of cement

Preinduction period

The duration of the preinduction period is only a few minutes, during and immediately following mixing. A large exotherm is observed at this time, resulting from the wetting of the powder and the rapidity of the initial hydration. From a physical standpoint, an initial layer of C-S-H gel is formed over the anhydrous C_3S surfaces.

*Induction period i*ndicates the induction period lasting several hours (hydration is almost halted). Dissolution of ions continues. The reason why hydration is inhibited here is not clear.

Acceleration period of C-S-H and CH form by crystallization of ions when Ca2+ and OH- ion concentrations super saturated in solution. When final products crystallized, hydrolysis of C_3S proceeds again rapidly (nucleation and growth control). The hydration products provide anchors on which more C-S-H can form, forming a coating, growing in thickness around the C3S grains. Whereas, it becomes difficult for H2O molecules to reach the anhydrous C3S and the production rate of hydration products decreasing, as it takes longer for H20 to diffuse through the coating as observed by who suggested that C-S-H clusters form by, “heterogeneous nucleation at the C_3S surface and then grow by accumulating C-S-H units

Acceleration and Deceleration Periods

At the end of the induction period, only a small percentage of the C3S has hydrated. The acceleration and deceleration periods, also collectively known as the “setting period,” represent the interval of most rapid hydration. During the acceleration period, solid $Ca\,(OH)_2$ crystallizes from solution and C-S-H gel deposits into the available water-filled space. The hydrates intergrow, a cohesive network is formed, and the system begins to develop strength. The porosity of the system decreases because of the deposition of hydrates. Eventually, the transportation of ionic species and water through the network of CS-H gel is hindered, and the hydration rate decelerates. At normal conditions, these events occur during couple of days. (Nelson,1990)[17]

Specifications for well cements were established by the API, because the conditions to which Portland cement is exposed in wells can differ radically from those experienced in construction applications. There are currently eight classes of API Portland cements as showed in Table 1. Classes are arranged according to the depths to which they are placed, and the temperatures and pressures to which they are executed.

API CLASS	ASTM TYPE	C3S	C2S	C3A	C4AF	Fineness (cm2/g)
A	I	45	27	11	8	1600
B	II	44	31	5	13	1600
C	III	53	19	11	9	2200
D	-	28	49	4	12	1500
E	-	38	43	5	9	1500
G	(II)	50	30	5	12	1800
H	(II)	50	30	5	12	1600

Table 1: API Cement Typical Phase Compositions

3.2 **Cement Additives**

Good bonding between cement and casing also between cement and rock formation are essential for effective zone isolation, hence usually the additive should be added to improve the characteristics of standard cement. Today, over 1000 additives for well cements are available, many of which can be supplied in solid or liquid forms. Seven categories of additives are generally recognized as shown below on Figure 6:

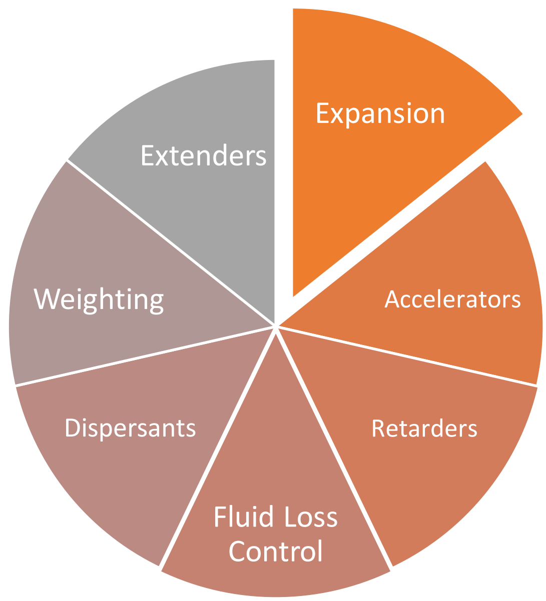


Figure 6: Classification of additives for well cementing

<u>Expansion agent:</u> One of the fundamental class of additives that affect the change in absolute volume are the expansive additive that must be controllable to produce the required expansion of the system at the right time. If crystallization occurs when the cement slurry is still in the fluid phase, the cement system is unable to sustain the expansive forces. For additives with very low reactivity, the crystallization pressure of expansive additive might result in cracking and loss of mechanical properties, so-called unsoundness and lead to carbonation shrinkage. Consequently, the reactivity of the additive must be designed somehow that its expansion occurs mainly in the late plastic phase and at the beginning of cement hardening phase. In the initial phase of hardening, sufficient anhydrous cement capable of hydration is still available for mutually recombining the particles of binding agent which have been driven apart by the crystallization pressure of expansive additive (Ghofrani and

Plack 1993)[18]. Therefore, CaO-type expansive additives cause expansion mainly at an early stage (Mo, Deng, and Wang 2016)[19]. MgO reactivity has been divided into three categories based on reactivity value, specific surface area (SSA) and agglomeration ratio (AR): highly reactive, medium reactive and less reactive (Jin and Al-Tabbaa 2013). These categories permit the use of MgO with appropriate reactivity according to the bottom hole conditions. Recently, there have been several studies: Mo, Deng, and Tang 2010, Mo et al. 2014, del Valle-Zermeno et al. 2012, Li et al. 2010, Tang et al. 2014, Salomao et al. 2014 on controlling MgO expansion properties through preparation and curing conditions However, still there is a lack of knowledge on expansion of MgO and CaO as a function of curing conditions specifically existence of free water pressure and temperature, which is essential for designing expanding oil well cement systems.

ACCELERATORS: chemicals which reduce the setting time of a cement system and increase the rate of compressive strength development. Many inorganic salts are accelerators of Portland cement. Among these, the chlorides are the best known, however, an accelerating action is also reported for many other salts including carbonates, silicates, specially sodium silicate, aluminates, nitrates, nitrites. sulfates, thiosulfates, and alkaline bases such as sodium, potassium and ammonium hydroxides. Accelerators are added to cement slurries to shorten the setting time, stages I and II of the hydration schemes described earlier in this Chapter and/or to accelerate the hardening process period III and IV. They are often used to offset the set delay caused by certain other additives, such as dispersants and fluid-loss control agents.

Retarders: chemicals which extend the setting time of a cement system. Like acceleration, the mechanism of set retardation of Portland cement is still a matter of controversy. Two principal factors must be considered: the chemical nature of the retarder, and the cement phase (silicate or aluminate) upon which the retarder acts. The major chemical classes of retarders are sodium and calcium salts of lignosulfonic acids, hydroxycarboxylic acids contain hydroxyl and carboxyl groups in their molecular structures, saccharide compounds so-called sugars, polysaccharides, alkylene phosphonic acids and their salts, boric, phosphoric, hydrofluoric and chromic, sodium chloride, zinc and lead oxides are widely used.

Fluid-Loss Control Agents: materials which control the loss of the aqueous phase of a cement system to the formation. When a cement slurry placed across a permeable formation under pressure, a filtration process occurs. The water-based slurry escapes in to the formation, leaving the cement particles behind.

Two principal classes of fluid-loss additives exist: finely divided particulate materials are bentonite and different types of water-soluble polymers.
Because of the complexity of the cement hydration process, and the large number of parameters involved, the only practical method for cement slurry design and avoiding unpleasant surprises at the wellsite is thorough laboratory testing before the job. It is essential that the tests be performed with a representative sample of the cement to be used during the cement job.
Weighting Agents: materials which increase the density of a cement system. When higher slurry densities are required, materials with a high specific gravity are added. To be acceptable as a weighting agent, such materials must meet several criteria. The most common weighting agents for cement slurries are ilmenite, hematite and barite.
Dispersants: chemicals which reduce the viscosity of a cement slurry. Well cement slurries are highly concentrated suspensions of solid particles in water. The solids content can be as high as 70%. The rheology of such suspensions is related to the supporting liquid rheology, the solid volume fraction and to interparticle interactions that depend primarily on the surface charge distribution. Cement dispersants adjust the particle surface charges to obtain the desired rheological properties of the slurry. Sulfonates are the most common cement dispersants.
Extenders: materials which lower the density of a cement system, and/or reduce the quantity of cement per unit volume of set product. Common cement extenders are bentonite, fly ashes, sodium silicates, microsphere and foamed cement that used to accomplish one or both of the following characteristics such as density reduction, yield-extenders, increasing of viscosity and preparation of foamed cement. (Nelson et al. 1990)[17]

Chapter 4
Technical evaluation of conventional cement failure

This chapter provides an explanation why often conventional cement does not withstand during with time and the reason behind that includes wrong job design, wellbore stability problem, fluid contamination, wrong slurry design and significant cement shrinkage.

4.1 **Purpose of cementing jobs**

The introduction of a cementitious material or cement slurries into the annulus between casing and formation or previous casing or both called cementing. The main purpose of cementing listed below in Fig.7 (Pine,2003)[22,23]

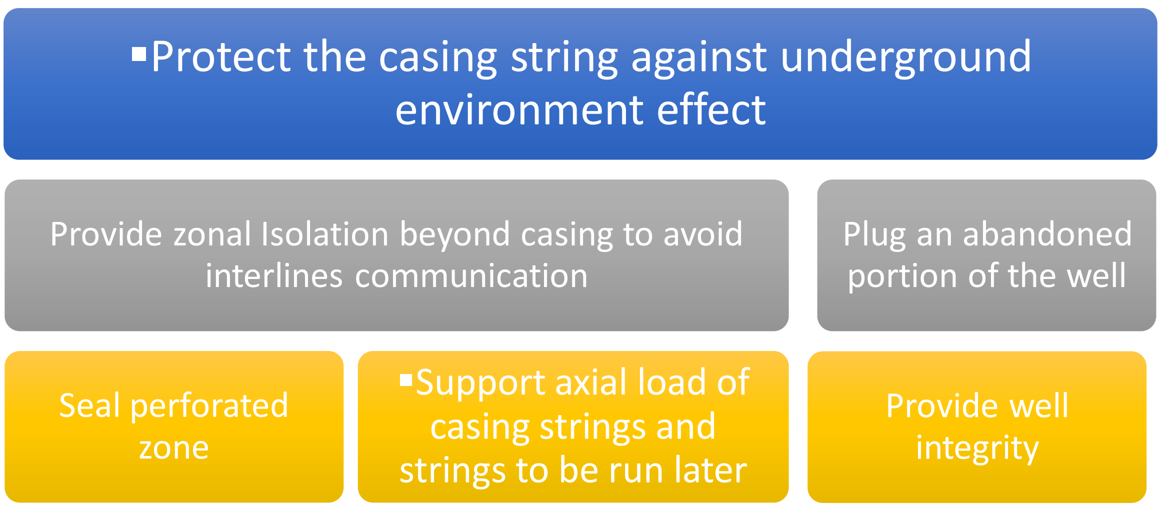


Figure 7: Purpose of cementing jobs

Thereafter if the cement sheath does not work effectively for purposes listed above the whole concept of well integrity will be fail. The quality of cement column is extremely important and proportional to production rate (Wilcox, 2016)[45]

.

4.2 **Contamination problem**

In order to reach a main goal of cementing the well must be cleaned from drilling mud, spacer and washes and the annular space must be completely filled with cement slurry. After cement slurry placed it must harden and develop the necessary mechanical properties for maintain a hydraulic seal during the life of the well. Whereas, good mud removal and proper slurry placement are essential to obtain zonal isolation. Incomplete mud displacement can leave a continuous mud channel across the zones of interest, thereby favoring interzonal communication and cement contamination. Bonding and cement seal durability are directly related to the efficiency of the displacement process. Therefore, mud displacement is crucial aspect shows ability of well cementing sheath sustain. However, drilling mud and cement slurry contact can occur due to bad hole cleaning that often lead to gel development of the slurry. When this happened drilling mud and the cement slurry are called incompatible. Incompatibility issue exists between fluids being displaced in the annulus, the displacing fluid such as the cement slurry that tends to channel through the viscous interfacial mass, leaving patches of contaminated mud sticking to the walls of the casing and formation. This may lead to insufficient zonal isolation and as a result cement failure. Contaminated cement slurry can also cause unacceptably high friction pressures during the cement job, with results in to possibility to fracture a fragile formation. In some cases, total plugging of the annulus can occur, preventing the completion of the cement job. To avoid such problems the rheological and chemical properties of mix fluids and hole cleaning must be carefully designed through contamination test. (Ravi and Xenakis, 2007)[20]

4.3 Shrinkage

When Portland cements react with water, the system cement plus water undergoes a net volume diminution. This is an absolute volume decrease. It occurs because the absolute density of the hydrated material is greater than that of the initial reactants.
The reduction of absolute volume that is known as shrinkage. If this occurs before the cement slurry settling it is called as plastic shrinkage. In case of volume reduction due to moisture loss the process known as drying shrinkage. In addition, to drying shrinkage, cement sheath settling can also experience volume reductions such as thermal contraction, autogenous shrinkage occurs as a result of the chemical reactions between molecules that take place during cement hydration. The hydration process represents schematically in Fig.8. As a result, the pore structure being partly emptied. The pressure is transferred to the solid and results in external/bulk contraction of the cementitious material (Lura, Jensen, and van Breugel 2003)[21]. Bulk shrinkage might lead to debonding and micro cracks of formation between the cement sheath and the casing or the formation, and/or tensile cracks lead to zonal isolation and fluid migration issue. (Ghofrani and Plack 1993)[18]. Magnitude of autogenous shrinkage is not significant in most of the material placed where shrinkage concerned.

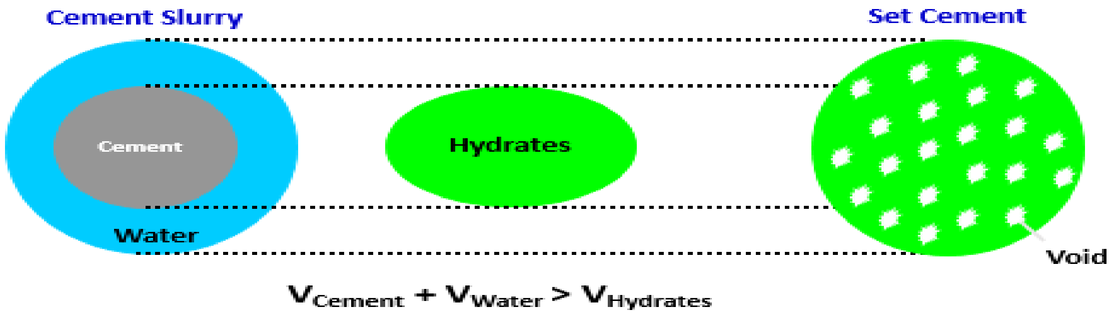


Figure 8: Schematic cement hydration process (Schlumberger, 2014)[2]

The majority cases cement shrinkage happens either in the plastic or the hardened state. In most well cementing operation, the likelihood of bulk and drying shrinkage is often greater than that of the other types of shrinkage mentioned above and can reach 1-3% of volume reduction.

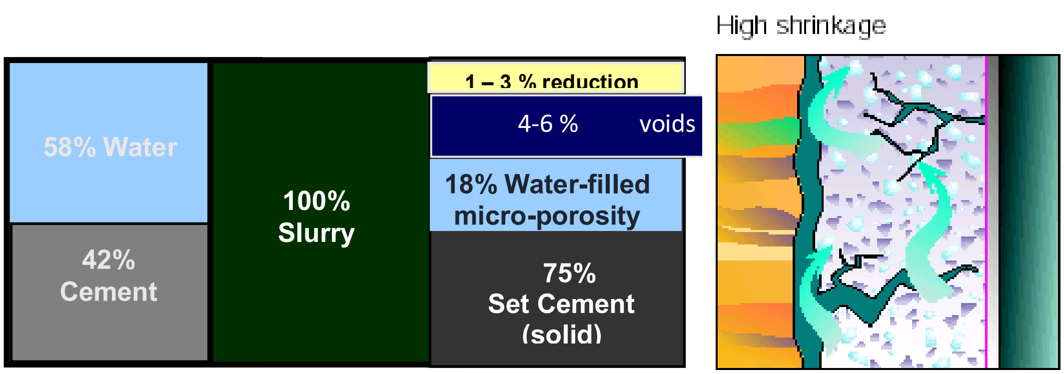


Figure 9: Representation of volume reduction of concreate (Schlumberger 2014)[2]

I worth case conducted from another study made by Lea 1971 the percentage of shrinkage can reached up to 8.7% depends from the chemical composition and curing parameters of Portland cement shows in Table 2.

Sample	1 Day	7 Days	28 Days	100 Days
#1 Portland cement	2.8	4.8	6.0	6.9
#2 Portland cement	1.7	4.4	-	6.3
#3 Portland cement	2.7	8.0	8.6	8.7
#4 without gypsum	2.6	6.3	7.5	7.6

Table 2: Percentage of absolute volume diminution of Portland Cement

4.4 **Well position**

Well deviation can affect cement slurry settling as cement sets differently in deviated wells and vertical wells non-equal stresses distributions around borehole lead to sheath failure. Drilling fluid is circulated in the annulus first to ensure that cuttings and borehole wall cavings have been removed before running the casing. Centralizers are then used to ensure that the casing is placed in the center of the borehole. For under-reamed or washed out holes, bow spring centralizers are used. After the cement slurry is pumped downhole, a lighter drilling mud follows. In this way, the casing is under compression from a higher differential pressure on the outside of the casing. Thus, when the cement sets and drilling or production operation continues, the casing will always have an elastic load on the cement-casing interface. According to Sweatman (2006) this load called also sustained casing pressures (SCP) and considered for maintenance bonding between cement and casing to prevent leakage and gas migration. In summary, the cementing problems that could cause SCP include: (1) micro annuli caused by shrinkage or expansion (2) channels caused by improper mud removal (3) lost circulation of cement into fractured formations (4) flow after cementing by failure to maintain an overbalance pressure (5) mud cake leaks (6) tensile cracks in cement matrix caused by temperature and pressure disturbance. (Wilcox, 2016)[45]

4.5 **Flash set**

When Portland cement grounded alone without gypsum and mixed with water the tricalcium aluminate rapidly reacts, the temperature markedly increases, and an irreversible stiffening occurs followed quickly by a pseudo-set. This phenomenon is called a "flash set," or sometimes a "quick set." The uncontrolled C3A hydration can be prevented by the addition of gypsum to the system. For optimum cement performance, the quantity of gypsum must be balanced according to the reactivity of the clinker. It is important to point out that a flash set can still occur if the quantity of gypsum in the cement is insufficient with respect to the reactivity of the clinker. Unfortunately, no simple rule exists to determine the optimum gypsum content, as this depends upon a variety of parameters, including cement particle size distribution, the alkalis and the aluminate phase content (Lerch, 1946)[41]

False set are reversible upon vigorous slurry agitation however, such agitation would not be possible during most well cementing operations, particularly if the slurry is mixed continuously. The addition of a dispersant can be useful for reducing the rheological impact of false sets with type of cement known to have such inclinations

4.6 **Aging cement**

The performance of Portland cement can be affected significantly by exposure to the atmosphere and/or high temperatures during storage in sacks or silos. The principal effects upon well cements include the following

- Increased Thickening Time
- Decreased Compressive Strength
- Decreased Heat of Hydration
- Increased Slurry Viscosity

The effects are principally due to carbonation of the calcium silicate hydrate phases, and partial hydration of the free CaO. The rate at which these processes occur is directly related to the relative humidity of the storage environment. The effects of limited cement exposure to air during transport operations have been shown to be less severe (Cobb and Pace, 1985)[42]. When Portland cement is stored in hot regions, the temperature in the silo can be sufficiently high to result in the dehydration of gypsum. Such cements would be more subject to exhibit the false-set phenomenon. Therefore, laboratory testing samples including all chemistry with correct batch numbers used must correspond to cement and additives from field storage.

4.7 **Mixing fluid**

The need for adequate workability to facilitate placement and consolidation of concrete often necessitates the use of a greater amount of liquid usually fresh water is needed for the hydration process. In the absence of fresh water, salt brackish water or seawater is frequently used for mixing cement slurries for offshore cementing operations. Such waters are advantageous because of their availability and economy. Brackish waters from lakes and ponds, vary significantly, and should be thoroughly tested in the laboratory prior to use on location.

Relative Laboratory testing has been identified the following effect of the sea water in Table 3 upon the performance of Portland cement slurry

- Reduction Thickening Time
- Increasing Fluid Loss
- Increasing Early Compressive Strength
- Slight Dispersing Effect
- Increasing Shear Bond Strength
- Increasing Slurry Foaming Tendency

	THICKENING TIME (HR:MIN)	COMPRESSIVE STRENGTH (PSI) AT 100 DEGF AFTER 24 HR
CEMENT WITH FRESH WATER	2:32	1780
CEMENT WITH SEAWATER	2:05	2150

Table 3: Thickening time and compressive strength of cement mixing with fresh water and seawater (Smith and Calvert,1974)

The most important species of water must be monitored are Cl^-, SO_4 Ca^{2+} Mg^{2+}, and various organic compounds resulting from the decomposition of plant material. Such impurities have significant effects upon the gelation or over retardation. All laboratory cement slurry design experimentation should be performed with a sample of the location water.

4.8 Sour environment

In sour gas environment, Portland cement is known to be thermodynamically unstable. It tends to strongly degrade when exposed into CO2-rich acidic conditions, by reacting with calcium hydroxide formed from hydrated calcium silicate phases. The one study has been done in order to indicate cement deterioration for that one of the greatest gas condensate fields in Pannonian basin (Croatia) was the motivation for research study carried out in order to investigate the possible corrosive mechanism. After almost 15 years of hydrocarbon production form naturally fractured carbonates, some wells begun to suffer of plugging across the perforated zone with unidentified debris, which was accompanied with water cut increase. That field characterize as a high reservoir temperature, BHST > 180°C, sour gas: 22% CO2 and 150 ppm H2S to the cement, as well as debris identification. The work indicated that oil well cement matrix under hostile downhole conditions after long-term exposure could be deteriorated loosing of compressive strength and its integrity. The dominant mechanism of cement deterioration is caused by CO2 corrosion process in the form of carbonic acid leaching (Krilov, Loncaric, & Miksa, 2000)[42]. In sour types environment Non-Portland based cements must be used which contain aluminum hydrates. An alternative is to use fortified Portland cement where resistance is increased by adding a latex diluent of a specific particle size and adding a non-standard, high alumina cement to reduce the amount of Portland cement. This has been used in the well completion design for acid gas where 65% hydrogen sulfide, 35% carbon dioxide had been injected over a 50-year period in Labarge area, Wyoming. Relevant cement additives that help to maintain well integrity include hydrazine which is used to control corrosion of the casing and radioactive tracers to assess where the cement has been placed. (Benge, 2005)[43]

Chapter 5 **Alternatives to conventional cement**

This chapter will cover possible alternatives to conventional cement as a sealing material. Two alternatives are be discussed in this chapter, are Magneto rheological blend cement (MRBC) and resin sealant technology. Before introduction into each technology the fundamental properties of a material for remediation operation would be established.

5.1 **Fundamental properties of sealant material**

The Materials which would qualify them as good candidate to be used for sealing well purposes must have certain capabilities:

- be capable of being placed at the appropriate depth
- be compatible with other fluid: Mud/Spacer/Formation fluids
- be sufficient structural strength to withstand loads
- be resistant to cracking and shrinking
- be able to form a tight bond with the borehole wall and the casing

The selection process of a sealing material must correspond to two main stages includes fluid behavior after placement, and also slurry properties that allowed fluid to be insert into the well. During placement slurry properties that necessary for a successful pumping job largely must be readjustable based includes the following main characteristics:

Rheology: The slurry must be sufficiently thin to be pumpable, however thick enough to such an extent that any entrained solids stay in suspension.
Pumping time: The length of time the material remains pumpable, so that it can be placed in the annulus properly. Normally, higher downhole temperatures make conventional cementitious materials set quicker than at lower temperatures, aggravating the pump time problem.
Transition time: cementing across high-pressure gas zones required rapidly slurry hardening occurrence, in order to minimize the time between loss of hydrostatic head and the strength development. During this transmission period and the strength development, gas can invade cement matrix, resulting loss of integrity.

Density: The density of the fluid must be readjustable. Low density can lead to big pressures difference between well annulus and formation that allowing the well to flow prematurely and result in loss of well control.
Contamination with other fluids: Contamination may result in lack of desirable properties, or the inability to seal annulus properly with slurry.
Moreover, to the slurry properties required while the fluid is pumping downhole, other properties are needed after the material has placed in the wellbore. Mechanical properties of the slurry at early stage will lead to the better well performance.
Development of compressive strength: Historically this was the primary aspect of strength that was considered in the design of cement slurries.
Development of tensile strength: Increasingly, it is being recognized that cement breaks in tension in the wellbore follow with cracking and the migration path generation. Tensile strength in Portland cement is low, and the construction industry compensates this by adding other ductile components such as iron particles. In oil and gas wells, reinforcement options are limited, so alternative material must have high levels of tensile strength.
Resiliency: This is an ability of the cement material to absorb stresses without failure. whereas, Portland cement is still brittle, so its ability to deform without failure is low. The ideal sealant material must be ductile enough to deform under load without failure
Shear Bond: sealant materials must bond not only to steel pipe but to a variety of formations encountered in the well. Bonding ability is the primary mechanism by which the material seals the required zone. (Edgley 2002)[44].
Conventional cement cannot correspond to all required properties listed above. Therefore, experimentation study plan must be evaluated precisely for different material to define the appropriate technology. Primarily, materials are used for this study must shows improvement in fundamental slurry properties compare to conventional cement. (Ravi, Bosma and Hunter, 2003)[23]

5.2 Magneto rheological blend cement (MRBC) System

MRBC technology has covered a novel technique to suspend a magnetorheological slurry, in a casing-by-casing annulus by eliminating gravitational migration of the fluid authorized by Well-Set company, Norway. This technique uses magnetorheological blend particles to form an effective barrier in an annular space for squeezing and for cross-sectional plug and abandonment (P&A) operations without cutting and pulling out casing.

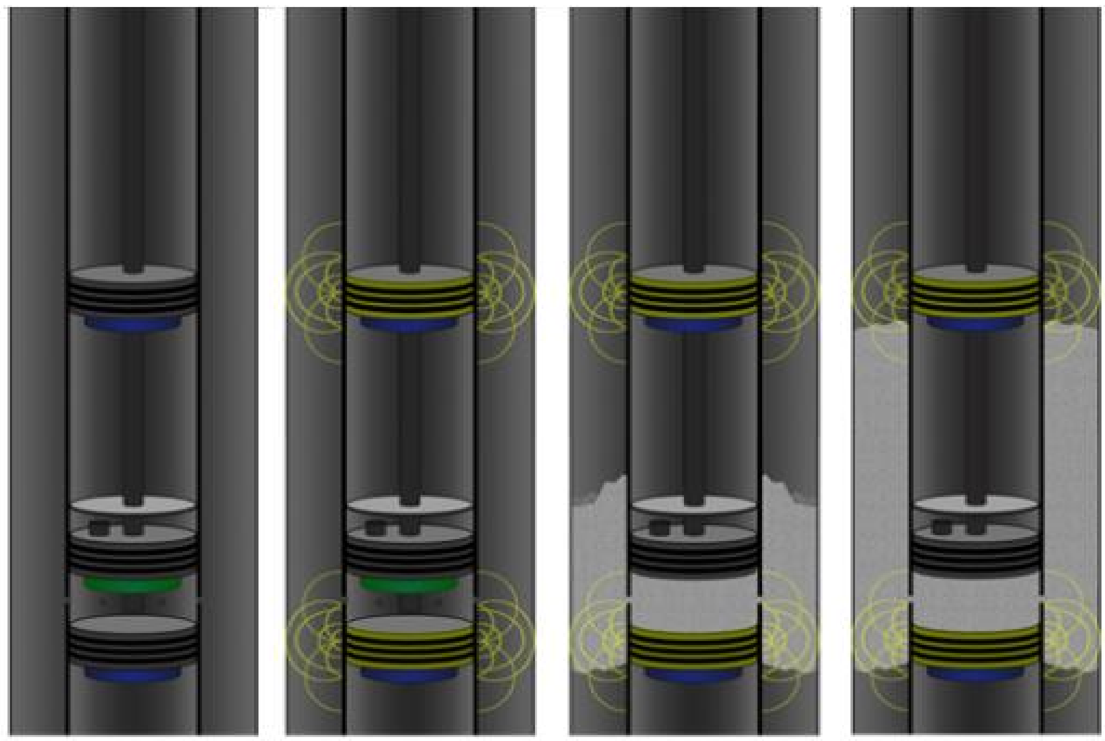


Figure 10: MRBC technique performance (Well-Set,2018)[25]

With MRBC technology Fig.10 we are sealing at least a portion of a well include a casing, a wellbore and an annulus between the casing and the wellbore. That allows casing annulus to be effectively plugged between defined depths using a dump bailer, a coiled tubing, a drill pipe, or any other type of cement providing means suitable for the purpose. Using a light intervention vessel is significantly less expensive than a drilling rig or drilling vessel. The MRBC is suspended between two magnetic fields until the slurry sets, meaning that there is no scope for gravitational slump. During squeezing of the slurry into the annulus region the magnetic field implies at switch on position lead to an effective bond with a surrounding casing and/or tubing and/or formation with no shrinkage, slurry sedimentation, channeling or slumping, and that it has reached its designed compressive strength.

The method may further comprise the step of:

- setting a base device in the first casing include bridge plug or an inflatable/swellable packer, or other device suitable for forming a physical base for a slurry.
- installing a first magnetic field inducing member in the well at a predetermined depth.

- installing a second magnetic field inducing member in the well at a predetermined depth, a distance above the first magnetic field inducing slurry height.
- stopping the magnetic field inducing means from inducing a magnetic field.
- testing the quality of the job when the MRBC has hardened and formed a sealed zone.

Well-Set are satisfied with using MRBC as a sealing material for P&A and Remedial operations and have qualified it for this purpose. (P27799PC00, Well-Set)[24,25]

5.3 Epoxy Resin System

Epoxy resin are increasingly being used in cementing remedial operation since they show excellent ductility and resistance to contaminations including oil based and water-based fluids, tailored mechanical properties, very low yield point, as well as availability solid-free design. (Jones, Watkins, White, 2015)[26]
Epoxy resin have a good rheological property and are more resistivity to sour environment comparing to conventional cement. Studies have proven that epoxy resins can enhance zonal isolation due to their ability to withstand differential pressure. (Foianini, 2014)[27]
Since they have low density, epoxy resin is more useful than conventional cement in areas where there is narrow fracture pressure gradient such as Deepwater Gulf of Mexico. Epoxy resin also provide excellent bonding in oil wet formation and enhance bond between the casing and slurry. (Al-Yami, 2017; London, 2013)[28,29]
Epoxy resin is an adhesive polymer containing epoxide group while curing agent is a chemical activator that reacts with an epoxy in specific concentration to produce a cross linked polymer. The initially modification of epoxy resin is transformed into a solid after reaction has been implemented. The reaction can be under a low curing temperature or high curing temperature.
Several factors affect the reaction between the hardener agent and the resin:

- Concentration and type of both the epoxy resin and the hardening agent will affect the settling time and the speed of the reaction. The concentration of each components needed for the reaction is calculated based on information about equivalent epoxy weight and hydrogen equivalent weight of curing hardener. (Garcia and Soares 2003)[31]
- Another factor affecting the reaction is temperature. The higher the temperature of curing conditions, the faster the reaction and stronger the final product.

- Third factor needed to be considered when curing is glass transition temperature which is the temperature above which the rein becomes brittle. Transition temperature depends on the flexibility between polymer molecules. As the molecule's flexibility increase, transition temperature decrease. To reduce this limitation of cured resin system, the resin was cured at temperature lower than transition temperature. The limitation for resin curing conditions is about 150 degC. (Fink 2013)[30].

Chapter 6 **Prove of concept (POC) experimentation**

Evaluating class of sealant material useful for remedial operations along with test procedures and experimentation results are discussed within this section. Experimentation work will focus on comparison study that differentiated by using different classes of material and slurry composition. In addition, influence of slurry contamination during placement will defined based on test outputs.

6.1 Test procedure and initial conditions

Three class of material chosen for initial study are conventional cement, MRBC and resin systems had cured at atmospheric pressure and temperature equal to 38 degC. Laboratory experimentation for each class were performed to determine the following system's parameters: rheology and free water, mechanical properties of pure and contaminated samples, sedimentation-shrinkage using API RP10B-2 standard.

6.1.1 Samples preparation and curing

Material's mixing procedure have been executed in the laboratory using standard API blade-type blender with 1-liter capacity. After mixing specific density slurry shall be heated up to 38degC. The maximum speed used during slurry preparation was 12,000 rotations per minute (rpm). The cement-based slurry must mix in the blender for 15 seconds at 4,000 rpm and 35 seconds at 12,000 rpm. Resin based slurry must mix under low shear rate condition less than 2000 rpm until the mixture is clear and homogeneous.

6.1.2 Rheological properties

The slurry must be condition at 38degC before obtaining the rheological measurements for 30 minutes. A rotational non-pressurized viscometer Fann Model-35 is used to measure the rheological properties of fluids. The rheological readings from 3 up to 300 rpm qualify for plastic viscosity and yield point calculation. Most cement slurries exhibit non-Newtonian behavior for which shear stress is not directly proportional to shear rate. For slurry density equal to 1.9SG plastic viscosity and yield point estimated may be within the range 20 - 100 cP and 10 - 35 lbf/100ft2.

6.1.3 Free water

The purpose of test from 6.1.3 and 6.1.4 is to determine the static stability of a cement slurry. The cement slurry is conditioned to simulate dynamic placement in a wellbore. The slurry is then left static to determine if free fluid separates from the slurry or to determine if the cement slurry experiences particle sedimentation. Free water can be formed with minimal sedimentation, and sedimentation can take place without free water being formed. Therefore, both the free water result and the sedimentation result are required in order to understand the static stability of the slurry under downhole conditions.

Excessive free water and sedimentation are considered detrimental to cement sheath quality. The amount of free fluid and sedimentation or both that is acceptable varies with the application. (API RP-10B2, 2013)[32]

When the cement slurry is allowed to stand for a period of time before setting, water may separate from the slurry. A free water test is used to measure the water separation using a 250 ml graduated cylinder in the cement slurry for two hours. This parameter is required to be zero or negligible due to ability of free water create a channel for wellbore fluids to flow specially for deviated wells.

6.1.4 Sedimentation test

Settling have been measured by comparing densities of different sections of the cement column cured. A cylindrically shaped cell, used to cure the cement slurry for the settling test, had a diameter of 1inch and length of 8 inch. Sections that were 2-inch-long were taken from different parts of the cement column sample. The cement slurry was cured at atmospheric pressure and 38degC for at least 24 hours. The density of each section of the cement was evaluated using an Archimedes principle. Homogeneous slurry solid particles distribution required for sedimentation test.

6.1.5 Compressive strength

Compressive strength measurement has been performed for pure and contaminated samples by crushing API cube molds using hydraulic press with the appropriate load rate on the controllers that refer to ASTM C109/C109M-07 after curing cubes for 24 hours at 38degC. Applying load is directly proportional to expected mechanical properties. If expected compressive strength is more than 500 psi, load rate must be 16,000 lbf +/- 1600 lbf per minute. If expected compressive strength is 500 psi or less, load rate must be 4000 lbf +/- 400 lbf per minute. The value of compressive strength calculated by dividing the force required to break the sample with the smallest measured cross-sectional area in contact with the load-bearing plates of the load frame. (Halliburton Cementing Technology Manual, 2007)[33]

6.2 Experimentation results

Conventional cement, MRBC and resin sealant were implemented into experimental matrix. Materials were conducted into study in order to define the best sealant class for remedial jobs application.

6.2.1 Conventional cement

To prepare 1.9SG conventional cement system Dyckerhoff class G-cement and fresh water were used. Figure 11 showed cured conventional cement sample that will follow with mechanical properties measurement

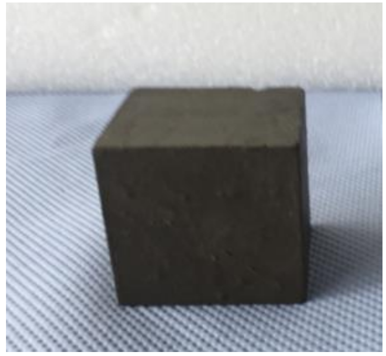


Figure 11: Cured conventional cement for compressive strength measurement

Composition

Job Type	**Remedial**	Depth	**not specified**	TVD	**not specified**
BHST	**48 degC**	BHCT	**38 degC**	BHP	**0 psi**
Starting Temp.	22 degC	Time to Bottom	03:00 hr:mn	Starting Pressure	0 psi
Slurry Density	**1.9 SG**	Slurry type	**Conventional**	**Mix Fluid**	**600 ml**
Code	**Concentration**	**Slurry volume**	**Component**	**Density**	**Supplier**
G cement	**792 ± 0.5 g**	**600 ml**	Cement	**3.2200 SG**	Dyckerhoff
Fresh water	**349 ± 0.5 g**		Base Fluid	**1.0 SG**	Tap water

Table 4: Composition of conventional cement slurry

Rheology

Temperature	38 degC		
(rpm)	Up (deg)	Down (deg)	Average (deg)
300	50.0	50.0	50.0
200	38.0	40.0	39.0
100	28.0	29.0	28.5
60	23.0	24.0	23.5
30	20.0	20.0	20.0
6	17.0	15.0	16.0
3	15.0	14.0	14.5
Rheo. computed	**Viscosity : 32.25 cP Yield Point : 17.8 lbf/100ft2**		

Table 5: Rheology of conventional cement slurry

Compressive strength

Slurry type	Compressive Strength value
Compressive strength after	**24hrs**
Conventional cement (CC)	**3938 psi**
95% CC + 5% Spacer/Mud	**2408 psi**
95% CC + 5% Diesel	**3717 psi**

Table 6: Compressive strength of conventional cement slurry

Free Fluid and Sedimentation

2.0 mL/250mL in 2 hrs
At 38 degC and 45 deg incl
Sedimentation : **1.2 %**

Table 7: Free Fluid and Sedimentation of conventional slurry

Figure 12: Cured conventional cement sample for sedimentation measurement

The existence of micro cracks, shrinkage, and stress cracking in cement slurry are several limitations that can negatively affect zonal isolation.
Experiment with 1.9SG conventional cement system shows high Free Water content equal to 2.0 ml and inappropriate solid particles distribution due to sedimentation content equal to 1.2% show in Table 7 that can lead to well issues during the life of the well, even if system performed good mechanical and rheological properties.

6.2.2 MRBC

To prepare 1.9SG MRBC system Dyckerhoff class G-cement, WellSet Iron Particles and fresh water were used. Figure 13 represent MRBC system placed into magnetic field which will follow with mechanical properties measurement after curing.

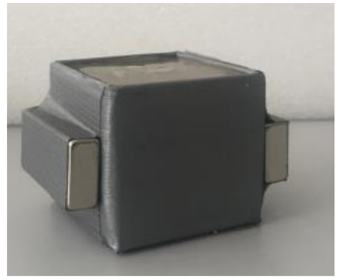


Figure 13: MRBC system placed into magnetic field

Composition

Job Type	**Remedial**	Depth	**not specified**	TVD	**not specified**
BHST	**48 degC**	BHCT	**38 degC**	BHP	**0 psi**
Starting Temp.	22 degC	Time to Bottom	03:00 hr:mn	Starting Pressure	0 psi
Slurry Density	**1.9 SG**	Slurry type	**magnetorheological**	**Mix Fluid**	**600 ml**
Code	**Concentration**	**Slurry volume**	**Component**	**Density**	**Supplier**
G cement	**760.3 g**	**600ml**	Cement	**3.2200 SG**	Dyckerhoff
Fresh water	**349 g**		Base Fluid	**1.0 SG**	Tap water
IP	**31.6 g**		Particles	**3.8 SG**	Wellset

Table 8: Composition of MRBC slurry

Rheology

Temperature	**38 degC**		
(rpm)	**Up (deg)**	**Down (deg)**	**Average (deg)**
300	**62.0**	**63.0**	**62.5**
200	**49.0**	**50.0**	**49.5**
100	**38.0**	**40.0**	**39.0**
60	**33.0**	**35.0**	**34.0**
30	**25.0**	**26.0**	**25.5**
6	**20.0**	**21.0**	**20.5**
3	**15.0**	**16.0**	**15.5**
Rheo. computed	**Viscosity : 35.25 cP Yield Point : 27.25 lbf/100ft2**		

Table 9: Rheology of MRBC slurry

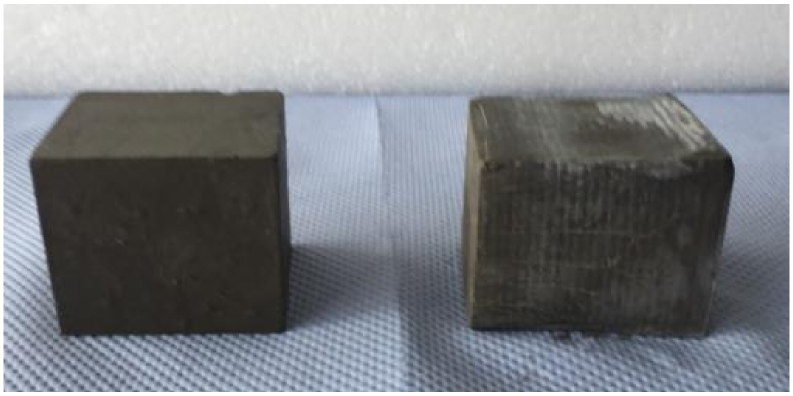


Figure 14: Cured MRBC system for compressive strength measurement

Compressive strength

Slurry type	Compressive Strength value
Compressive strength after	**24hrs**
MRBC	**2064 psi**
95% MRBC + 5%Spacer/Mud	**1175 psi**
95% MRBC + 5% Diesel	**1717 psi**

Table 10: Compressive strength of MRBC

Free Fluid and Sedimentation

0 mL/250mL in 2 hrs
At **38 degC** and 45 deg incl
Sedimentation : 0.03 **%**

Table 11: Free Fluid and Sedimentation of MRBC slurry

While using MRBC system is successful in several field application, there is still some limitation such as sharp decrease of compressive strength Table 10 due to iron particles distribution within magnetic field and incompatibility with drilling fluid caused micro cracks within cement matrix.

6.2.3 Resin system

Two components-based liquid samples were used for modified low viscous XYZ resin preparation. Figure 15 represented XYZ resin system sample after curing at 38 degC for 24 hrs.

Figure 15: Cured resin system for compressive strength measurement

Composition

Job Type	**remedial**	Depth	**not specified**	TVD	**not specified**
BHST	**48 degC**	BHCT	**38 degC**	BHP	**0 psi**
Starting Temp.	22 degC	Time to Bottom	03:00 hr:mn	Starting Pressure	0 psi
Slurry Density	**1.1 SG**	Slurry type	**resin**	**Mixture type**	**liquid**
Code	**Concentration**	**Slurry volume**	**Color**	**Density**	**Provider**
Component A	**1**	**1000ml**	Pale yellow	**1.1 SG**	
Component B	**1**		Clear	**1.0 SG**	

Table 12: Composition of resin sealant

Rheology

Temperature	**38 degC**		
(rpm)	**Up (deg)**	**Down (deg)**	**Average (deg)**
300	**208.0**	**208.0**	**208.0**
200	**145.0**	**145.0**	**145.0**
100	**75.0**	**74.0**	**74.5**
60	**48.0**	**49.0**	**48.5**
30	**27.0**	**26.0**	**26.5**
6	**5.0**	**5.0**	**12.5**
3	**3.0**	**3.0**	**3.0**
Rheo. computed	**Viscosity : 200.25 cP Yield Point : 7.75 lbf/100ft2**		

Table 13: Rheology of resin sealant

Using hydraulic press Fig.16, the compressive strength of the XYZ resin sealant system was measured with results shown in Table 14.

Compressive strength

Slurry type	Compressive Strength value
Compressive strength after	**24hrs**
XYZ resin	**12527 psi**
95% XYZ + 5%Spacer/Mud	**5182 psi**
95% XYZ resin + 5% Diesel	**11130 psi**

Table 14: Compressive strength of MRBC

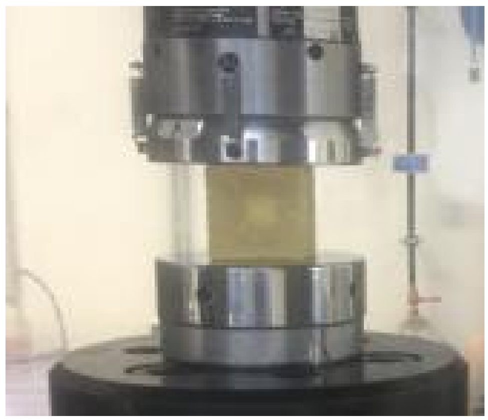


Figure 16: Hydraulic press for compressive strength measurement

Tests were done using contaminated samples of the system after curing material up to 38 degC. One of the tests was done with XYZ resin slurry contaminated with 5% spacer/mud mixture gives visible sample volume enlargement of the system from initial volume due to fast chemical reaction between active component as has showed on Fig. 17. and Fig. 18. But the nature and chemistry of the reaction must be studied and well understood before claiming this sample as expanded.

Figure 17: Cured resin slurry contaminated with 5% spacer/mud

Figure 18: Cured resin slurry contaminated with 5% spacer/mud

From the other hand cured 100% resin sample shows negligible visual shrinkage due to greasing mold surface as shown on Fig. 19.

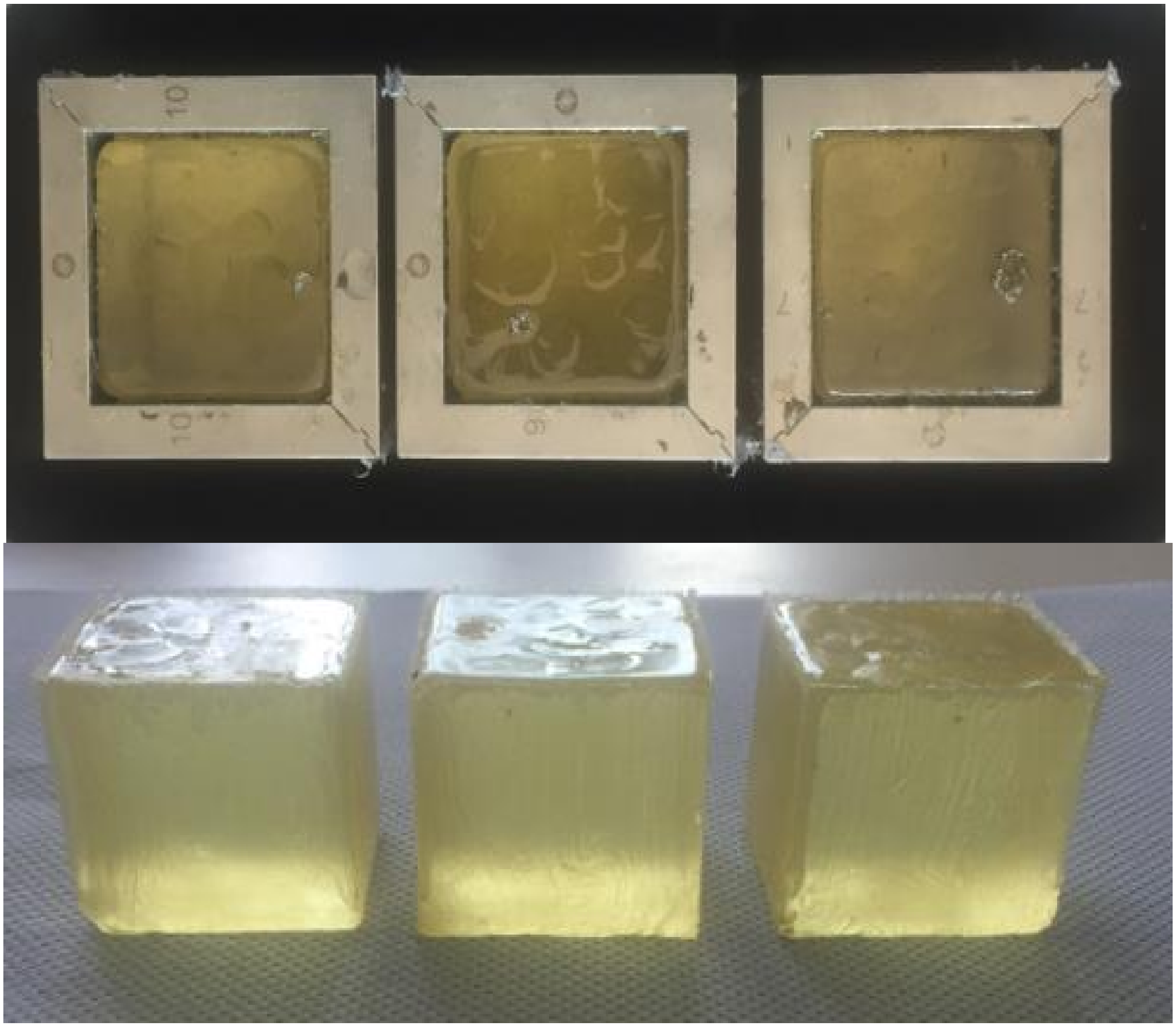


Figure 19: Cured pure resin slurry

The same time resin slurry contaminated with 5% diesel mixture slightly different in color changes from yellow to white and not changed initial slurry volume as shown on Fig. 20.

Figure 20: Cured resin slurry contaminated with 5% diesel

XYZ resin shows good rheological properties Table 13 and high compressive strength Table 14. And shows more resistive to slurry contamination, even shows visible sample volume enlargement due to spacer contamination that must require further investigation. Free Fluid and Sedimentation for resin system were not performed due to absence of water and particles distribution on the system. Prove of concept experimentation have evaluated that XYZ resin can be studied further as enhance zonal isolation material due to their ability to excellent bonding to formation/casing. While resin system is successful in several parameters, there is still some limitation such as temperature stability and drilling fluid compatibility that must be studied more precisely based on bottom hole temperature and pressure condition in remedial operation.

Conclusion

For evaluating alternative cement material for remedial operation, the different systems were executed into initial prove of concept study. This work defines optimum class of material that will be used according to further case study. Whereas, conventional cement slurry was used as a reference system. MRBC technology was suggested by WellSet company as novel technique that can overcome conventional cement system. And finally, liquid XYZ resin systems as a system that increasingly used in cementing remedial operation since it gives excellent results based on literature review. Experimentation work has proven that the XYZ resin system shows outstanding performance that will follow with next conclusions:

1. Resin system have better mechanical properties than conventional cement
2. Resin system provide enhanced zonal isolation due to better bonding capacity than conventional cement and MRBC
3. Resin system is adjustable to specific downhole conditions by changing their components concentration
4. Resin system is easy to density control than conventional system since is solid free system
5. Curing time can be modified for specific downhole conditions by adding retarder or accelerator additives

Further recommendation for this thesis will include the follow instructions:

- industry real case study implementation includes specific well information like downhole condition, formation details and possible contamination issue
- additional XYZ resin testing under varied conditions to ensure successful remedial operations.
- different from XYZ resin types of resin testing that may reveal a better performance for a real case.

References

[1] ISO/TS 16530-2, Well integrity Part 2: Well integrity for the operational phase, 2014
[2]Schlumberger, Presentation Primary-Cementing-Overview-n-Casing-Hardware 2014
[3]API STD 53 : Blowout Prevention Equipment Systems for Drilling Wells, 4th edition November 2012
[4] Investigation annual report summary from PSA, 2017
[5] NORSOK standard D-010, Well integrity in drilling and well operations". rev 3, 2004.
[6] NORSOK standard D-010, Well integrity in drilling and well operations, rev 4, 2013
[7] Cameron manifold AWT Why is well integrity good business, Pesa News, 2010
[8] Eventogo, Effective Well Integrity Management along WIF Guideline, 2012. http://www.eventogo.com/event/effective-well-integrity-management-along-wif- guideline
[9] Petroleum Safety Authority Norway, The Activities Regulations, 2018.
[10] RISK LEVEL IN THE NORWEGIAN PETROLEUM ACTIVITY SUMMARY REPORT, NORWEGIAN CONTINENTAL SHELF PETROLEUM SAFETY AUTHORITY NORWAY, TRENDS 2016
[11] An Introduction to Well Integrity, Norwegian University of Science and Technology, Rev4 December 2012
[12] Duijm, N. J. and Markert, F, Safety barrier diagrams as a tool for modelling safety of hydrogen applications. International Journal of Hydrogen Energy, 2009
[13] Duijm, N. J., Safety barrier diagrams as a safety management tool. Reliability Engineering and System Safety, 2009
[14] Perricone, M. J. Opportunities to Improve Oil and Gas Well Barrier Stewardship. RJ Lee group Inc, 2012
[15] Dickson Udofia Etetim, Well Integrity behind casing during well operation. Alternative sealing materials to cement. Master's Thesis at the Norwegian University of Science and Technology, December 2013
[16] Stuart,C., Wright,S and Foo,S. Application of an intelligent system to ensure Integrity throughout the entire will life cycle, SPE 135907, 2010.
[17] Nelson, E.B.: "Well Cementing," Development in Petroleum Science, Vol. 28, 1990
[18] Ghofrani R, Plack H, CaO- and/or MgO-Swelling Cements: A Key for Providing a Better Annular Sealing?. Conference Paper SPE-25697-MS, 1993
[19] Mo, Deng, and Wang Nano-Sized MgO with Engineered Expansive Property for Oil Well Cement Systems, Conference Paper SPE 180038-MS, 2016
[20] Ravi, K and Xenakis, H.: "Cementing Process Optimized to Achieve Zonal Isolation". Halliburton, January 2007

[21] Lura, Jensen, and van Breugel, Autogenous shrinkage in high-performance cement paste: An evaluation of basic mechanisms. Delft University of Technology, CEMENT AND CONCRETE RESEARCH Journal publication 2003
[22] Pine, M., Hunter, L., Mutch, J., Adam, J., and Griffith, E.J.: “Selection of Foamed Cement for HPHT Gas Wells Proves Effective for Zonal Isolation-Case History,” paper SPE 79909, SPE Drilling Conference, Amsterdam, Netherlands, February 2003
[23] Ravi, K., Bosma, M., and Hunter, L.: “Optimizing the Cement Sheath Design in HPHT Shearwater Field,” Conference Paper SPE 79905, SPE Drilling Conference, Amsterdam, Netherlands, February 2003
[24] Wellset As. Corporation Web Site. [Online]: http://www.wellset.no/home
[25] Patent number P27799PC00, Well-Set company ,2018
[26] A.R. Jones, C.A. Watkins , S.R. White , N.R. Sottos, Self-healing thermoplastic-toughened epoxy, Article published 2015
[27] Foianini, Frisch, Jones, Successful Identification and Bond Assessment of Epoxy-Based Resin Cement Behind Production Casing: Integrating Cementing Technology With New Log Interpretation Methodology to Provide an Innovative Well Integrity Solution. Conference Paper SPWLA-2014-HHH, 2014
[28] Al-Yami An Overview of Polymer Resin Systems Deployed for Remedial Operations in Saudi Arabia, SPE-188122-MS, 2017
[29] London B., Tennison B., Karcher J., Jones P.,Unconventional Remediation in the Utica Shale Using Advanced Resin Technologies, SPE 165699-MS Conference Paper, 2013
[30] J. Fink, Reactive Polymers Fundamentals and Applications: A Concise Guide to Industrial Polymers, A Concise Guide to Industrial Polymers, Second Edition, 2013
[31] F.G. Garcia, B.G. Soares, Determination of the epoxy equivalent weight of epoxw resin based on diglycidyl ether of bisphenol Aby proton nuclear magnetic resonance, 2003
[32] API RP 10B-2/ISO 10426-2, Recommended Practice for Testing Well Cement, April 2013
[33] Halliburton Energy Services: “Halliburton Cementing Technology Manual”, 2007
[34] Birkeland, S.T., Well Integrity of Subsea Wells during Light Well Interventions. Masters Thesis at the Norwegian University of Science and Technology, June 2005
[35] Birkeland,F.Final Field Permanent Plug and Abandonment – Methodology Development, Time and Cost Estimation, Risk Evaluation - Master thesis University of Stavanger, 2011
[36] Braune, H., Maintaining well integrity during slot recovery operations. Master’s degree thesis work at the department of Earth Sciences and Petroleum Engineering, Norwegian University of Science and Technology, NTNU, Norway, 2012

[37] The Activities Regulations. The Petroleum Safety Authority Norway, the Environment Agency, the Directorate of Health, the Labor Inspection Authority, 2016
[38] Schlumberger. Oilfield Glossary. http://www.glossary.oilfield.slb.com/. Accessed 2012.
[39] Ersdal, G., 2014. Safety of Structures, Compendium at University of Stavanger, 2014
[40] Lauridsen, O., Lootz, E., Husebo, T., Ersdal, G., Barrier management and the interaction between technical, operational and organisational barrier elements, Article in SPE International Conference and Exhibition on Health, Safety, Security, Environment and Social Responsibility, SPE-179364-MS, SPE 2016
[41] Lerch, W.: Portland Cement,1946
[42] Cobb, J. A. and Pace, R. S.: "Elements Affecting the Thickening Time of a Cement Blend," Conference Paper SPE 14195, 1985
[42] Krilov Z., Loncaric B., Micsa Z., Investigation of a Long-Term Cement Deterioration Under a High-Temperature, Sour Gas Downhole Environment Conference Paper SPE 58771-MS, 2000
[43] Glen Benge, Overview: Cementing Technology, Conference Paper ,SPE-0805-0040-JPT, 2005
[44] Edgley, K., D., Sabins, F., L. and Watters, L., T., Supercement for Annular Seal and Long Term Integrity in Deep, Hot Wells, Phase II Annual Report, CSI Technologies, Houston. August 2005
[45] B. Wilcox , B. Oyeneyin, S. Islam, HPHT Well Integrity and Cement Failure, Robert Gordon University, Conference Paper SPE -184254-MS, 2016