

UNIVERSITI TEKNOLOGI PETRONAS

# FINAL YEAR PROJECT II

# **Final Report**

# **CEMENT SLURRY DESIGN FOR SQUEEZE CEMENTING SOLUTION PROVIDE ANNULAR ISOLATION IN DUYONG FIELD MALAYSIA**

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# **CERTIFICATE OF APPROVAL**

# CEMENT SLURRY DESIGN FOR SQUEEZE CEMENTING SOLUTION PROVIDE ANNULAR ISOLATION IN DUYONG FIELD MALAYSIA

By

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# TRONOH, PERAK JULY 2011

# **CERTIFICATION OF ORIGINALITY**

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

AMIR FAIZ MOHD NAZIR

## Abstract

Among the main problem during cement squeeze operation is the fluid loss to the formation and the low compressive strength of set cement. During cement squeeze operation, the cement slurry will be force into the formation by pressure. This phenomenon will create differential pressure between the cement slurry and the formation thus will lead to the high filtration where lot of fluid inside the cement slurry will filtrate out. As the result set cement will bridge off and not set properly inside the permeable formation thus resulting in failure of squeezes cementing operation.

The objective of this project was to create cement slurry design which focusing on creating high compressive strength cement and low fluid loss cement slurry. Critical studies had been carried out to understand and identify the suitable additives that give high influence on those parameters. The expected cement slurry design could also be utilized at Duyong's field Malaysia as this cement slurry will be focusing to meet the criteria of Duyong's well.

In the early stage of this project there has been literature review studies about the others research which had been done before to eliminate the redundant of the research. Then the research was continued with the understanding on conventional cement which was Portland Type G oil well cement. Next further studies has been emphasizing on the additive which was suitable to meet the objective. The sample of the cement has also been tested at laboratory by using the equipment of Compressive Strength Tester and High Pressure and High Temperature Filter Press in order to evaluate the properties.

Prior to the requirement of Duyong field, Malaysia, the expected result for this project was to create the set cement slurry with high compressive strength of 22MPa and low fluid loss cement slurry of 15cm<sup>3</sup>/30min.

As the conclusion, this project has been successfully create a cement slurry design with high compressive strength set cement and low fluid loss cement slurry. These two parameters were really crucial in squeeze cement operation and will help in increasing the success rate of the operation.

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## Nomenclature

- $_{A^2}$  =Area through which filtration occurs (cm<sup>2</sup>)
- $V_{filtrate}$  = Filtrate volume (or fluid loss volume (ml)
  - $t = \text{Time}(\min)$
  - $k_z$  = Permeability of elementary layer at height z (uD)
  - u = Filtrate viscosity (cp)
  - $\Delta P$  = Differential pressure across the elementary layer (psi)
  - $R_z$  = Cake volume settled by filtrate volume unit at height z (cm<sup>3</sup>)
  - $\sigma$  = Compressive strength (psi)
  - F = Force (lb)
  - A = Area of set cement (inch<sup>2</sup>)
  - Q'f = API calculated filtrate rate (ml/30min)
  - Qf = Raw filtrate from experiment (ml)
  - Ws = Weight of silica fumes (lb)
  - X = Percentage of silica fumes (%)
  - Wc = Weight of solid cement (lb)
  - Vs = Volume of silica fumes (gal)
  - $\rho s$  = Density of silica fumes (ppg)
  - Wca= Weight of calcium chloride (lb)
  - Y = Percentage of calcium chloride (%)
  - Ww = Weight of water (lb)
  - Vca = Volume of calcium chloride (gal)
  - $\rho ca$  = Density of silica fumes (ppg)
  - Wa = Weight of additive (lb)
  - B =Gallon per sacks (ghs)
  - $\gamma$  = Specific gravity of additive

Va = Volume of additive (gal)

 $\rho a$  = Density of additive (ppg)

Ww = Weight of water (lb)

C = Ratio of water (%)

*Vw* = Volume of water (gal)

 $\rho w$  = Density of water (ppg)

*Wt* = Total weight of slurry (lb)

*Vt* = Total volume of slurry (gal)

 $\rho t$  = Density of slurry (ppg)

# CHAPTER 1 INTRODUCTION

## 1.1 Background of Study

Duyong gas field was offshore field and located approximately 220km of east Peninsular Malaysia. The water depth was ranging from 70 to 80 meters which measured from mean sea level. The first gas was produced in 1984 and the complex comprises of three drilling platform which were DDP-A, DDP-B and DDP-C, a central processing platform (CPP), a gas compression platform (GCP), a flare tripod (FT), and a living quarters platform (LQP). Gas has been sent to Peninsular Malaysia gas terminal through underwater pipe<sup>[1]</sup>.

Duyong shallow gas second mitigation studies indicated that well B-4 had fair-topoor cement bond behind the 9 5/8-in. casing. Temperature logs recorded in 2002 which gave positive indication of fluid movement behind the casing and the study also explained that the shallow "R" reservoirs likely contributed to the shallow gas problem. As a result, Duyong B-4 was selected as workover candidate in 2003. Petronas Carigali Sdn. Bhd. undertook the challenge to perform block squeezing above the top producing sand <sup>[1]</sup>.

Block squeezing was referred as to perforate above and below the pay section and then squeeze cement through the perforation. There were several purpose of this block squeezing such as to control high GOR by isolating the oil zone from an adjacent gas zone, to control excessive water or gas, to repair casing leak, to seal off thief zone, to isolate zone in permanent completions and to prevent fluid migration from abandoned zones <sup>[2]</sup>.

In order to achieve excellent result for block squeezing operation, optimize cement slurry design should be understand analytically and practically. This research was provided to study on the optimize cement slurry design which specialized on the creating of low fluid loss cement slurry and high compressive strength cement.

## **1.2 Problem Statement**

## 1.2.1 Problem Identification

Prior to perform squeeze cementing operation, it is very important to control the filtration in the cement slurry. This is because loss of filtrate through a permeable medium will cause a rise in slurry viscosity and a rapid deposition of filter cake which will restrict the flow. The compressive strength of the cement also plays a major part in squeeze cementing operation. The set cement should have high strength in order to withstand pressure inside the formation.

## 1.2.2 Significant of the Project

Through this project, high compressive strength cement and low fluid loss slurry has been design which will be optimized from conventional cement slurry. These two parameters really crucial in squeeze cementing operation and by developing this cement slurry design, it could be used at Duyong field, Malaysia.

## **1.3 Objective**

- To optimize the filtration control in cement slurry for squeeze cementing.
- To optimize the compressive strength of the cement.
- To determine the additive for cement slurry design suitable for Duyong's field.

### 1.4 Scope of Study

The scope of study was mainly on creating optimized cement slurry design in creating cement slurry which was having low fluid loss and high compressive strength. Parameters on the Duyong sand layer has been carried out in order to create the properties for the cement slurry. Further understanding in several additives for cement slurry has also been undertaken for achieving the objective. Lot of laboratory experiment has been conducted to create this optimize cement slurry.

## 1.5 The Relevancy of the Project

Cement squeeze operation will be very beneficial and cost efficient if successful. This is because this job will help during secondary recovery as helping creating zonal isolation, repairing casing leak, sealing off thief zones, correcting a defective primary cementing job and etc.

## 1.6 Feasibility of the Project

This project was encompassing research and laboratory work. Most of equipment and material were already available at Drilling Fluid Laboratory which is under Geoscience & Petroleum Engineering Department. This project has been done within 8 months. The objective has been achieved by following tight schedules and extensive studies.

# CHAPTER 2 THEORY AND LITERATURE REVIEW

#### 2.1 Theory

Several factors need to be fully understood in order to develop low fluid loss cement slurry with the high compressive strength of cement.

## A) Filtration Control

Filtration control or fluid loss control is the act of controlling (usually lowering) the volume of filtrate that passes through a filter medium. Control of fluid loss for a mud is achieved by several means, one of which is by addition of fluid-loss-control materials to the slurry system. Another is to change the slurry chemistry to make the materials already present work better. Adding a clay deflocculant to freshwater slurry typically improves fluid-loss control <sup>[3]</sup>.

There are lot of parameter need to be considered under filtration control such as permeability of the cake or the formation, differential pressure and length of time the differential pressure is maintained. In order to cope with this parameter the additives is introduced into cement composition to alter the properties of the slurry. Two most widely used filtration control additives are organic polymers (cellulose) and friction reducers. The theory behind these additives is to form films which will control the flow of water from the cement slurry and prevent rapid dehydration. The second one is to improve particle size distribution which determines how liquid is held or trapped in the slurry. The high molecular weight cellulose compound will produce low water loss in all types of cementing composition at concentration from 0.5 to 1.5 wt% of cement whereby the friction reducer are commonly added to cement slurry to control filter loss by dispersing and packing the cement particles and thus densifying the slurry<sup>[2]</sup>.

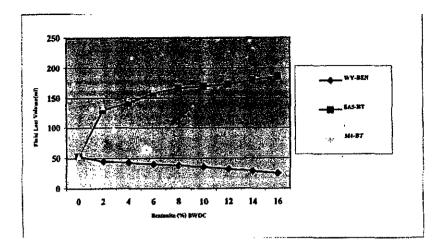


Figure 1. Fluid Loss of Cement Slurry with Various Amount of Untreated Bentonite at Room Condition<sup>[4]</sup>

There have been extensive studies on the filtration analysis by J, Desbrieres and the outcome is the numerical analysis for filtration control. Equation 1 is the numerical formula to determine the filtration volume<sup>[5]</sup>.

In this formula several factors seem to give influence on the filtration rate which was the permeability, core area, differential pressure, fluid viscosity, time and filtration cake. API has indicated that the Equation 2 and 3 were supposed to be used in order to find the filtration loss for laboratory test <sup>[6]</sup>.

a) Case with filtration burst

$$Q'f = \frac{2Qf(5.477)}{\sqrt{t}}$$
 (2)

b) Case without filtration burst

$$Q'f = 2Qf.....(3)$$

Several advantages of low fluid loss of cement slurry had been identified in squeeze cementing operation such as reduces premature dehydration in tubing and casing while squeezing perforation, satisfactory squeeze result at low pressure without over displacing, high pressure squeezing by hesitation technique with filter cake build up in perforation and help protect water sensitive shale section that may weaken and breakdown due to cement filtrate <sup>[2]</sup>.

#### B) Compressive Strength

Maximum stress a material can sustain under crush loading. The compressive strength of a material that fails by shattering fracture can be defined within fairly narrow limits as an independent property. In other hand, the compressive strength of materials that do not shatter in compression must be defined as the amount of stress required to distort the material an arbitrary amount. Compressive strength is calculated by dividing the maximum load by the original cross-sectional area of a specimen in a compression test <sup>[7]</sup>.

Type of cement is the major influence on the cement compressive strength. Down hole parameters such as temperature and pressure also give effect to the compressive strength as theses two parameters involve vitally during hydration of cement. Besides, water content, admixes and stirring time also give effect to the compressive strength of the cement. The theory behind compressive strength starts during static condition when gel strength takes places very rapidly within cement slurry. Gel strength development is a by product of the hydration process and signals the point which the cement slurry starts its change from a true hydraulic fluid that transmits full hydrostatic pressure to a solid set material that has measureable compressive strength. During this phase the cement slurry continually gain strength which enables a potential pressure restriction to occurs in the cement filled annulus <sup>[2]</sup>.

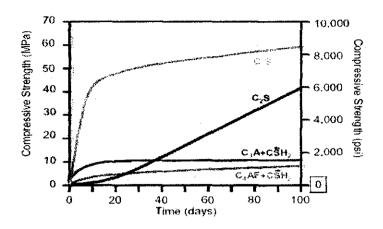


Figure 2. Graph Showing Compressive Strength Development in Paste of Pure Cement Compund<sup>[8]</sup>

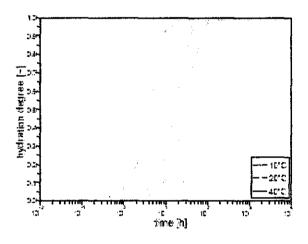


Figure 3. Influence of Temperature on Cement Hydration<sup>[9]</sup>

As for the numerical analysis, a compressive strength formula has been formulized. Equation 4 is the numerical formula in determination of compressive strength <sup>[7]</sup>

$$\sigma = \frac{F}{A} \tag{4}$$

The determination of compressive strength is based on the parameter of force and area of set cement.

## 2.1.1 Type of Cement

The cement type are characterize according to the API classification as published in API Standards 10, "Specification for Oil-Well Cement and Cement Additives."

Table 1: The Difference Classes of API Cement for Use at Downhole Condition <sup>[2]</sup>.

API	Mixing Water	Slurry	Well Depth	Static
Classification	(gal/sack)	Weight	(ft)	Temperature
		(lbm/gal)		(°F)
A (Portland)	5.2	15.6	0 to 6000	80 to 170
B (Portland)	5.2	15.6	0 to 6000	80 to 170
C (high early)	6.3	14.8	0 to 6000	80 to 170
D (retarded)	4.3	16.4	6000 to 12000	170 to 260
E (retarded)	4.3	16.4	6000 to 14000	170 to 290
F (retarded)	4.3	16.2	10000 to 16000	230 to 320
G (basic)	5.0	15.8	0 to 8000	80 to 200
H (basic)	4.3	16.4	0 to 8000	80 to 200

## 2.1.2 Additive in Cement Slurry

The inventions of basic cement which are API Classes G and H have allowed the use of additives become more flexible. Cement slurries can be tailored for specific well requirement around the world. Practically all cement additives are in form of free flowing powders that been sold by the provider.

## 1) Cement Accelerators

Cement slurries which will be used at shallow and low temperature would require acceleration to shorten thickening time and to increase early strength.

Table 2. Common Accelerator in Cement Slurries<sup>[2]</sup>

Amount Used ( wt% of Cement)
2 to 4
3 to 10
20 to 100
1 to 7.5
0.5 to 1.0

# 2) Lightweight Additives

When prepared from the API Class A, B, G, or H cement using the recommended amount of water, the cement slurry will weight excess than 15 lbm/gal. These additives would then be required to reduce the weight of the slurry. The additives also make slurry cheaper, increase yield and sometime lower filter loss. <sup>[2]</sup>

Table 3. Common	Lightweight	Additive in	Cement Slurries <sup>[2]</sup>
		1 100 Million ( V III	

2 to 16 wt% of Cement
1 to 50 lbm/sack of cement
5 to 50 lbm/sack of cement
5 to 20 lbm/sack of cement

Nitrogen	0 to 70 wt% of Cement

## 3) Heavyweight Additives

To overcome high pressure encounter in deep well, cement slurries of high density would be required. This additives should have specific gravity in the range of 4.5 to 5.0, low water requirement, not significant reducing cement strength, very little effect on pumping time, exhibit a uniform particle size, chemically inert and not interfere with well logging <sup>[2]</sup>.

Table 4. Common Heavyweight Additive in Cement Slurries<sup>[2]</sup>

Amount Used ( wt% of Cement)
4 to 104
5 to 100
10 to 108
5 to 25

## 4) Cement Retarder

As prior to prevent the cement from setting too quickly, retarders would require to be added in cement slurry. Retarder must be compatible with the various additives used in cement as well as with the cement itself.

# Table 5. Common Retarder Additive in Cement Slurries<sup>[2]</sup>

Amount Used ( wt% of Cement)
0.1 to 1.0
0.1 to 2.5
0.1 to 1.5
14 to 16 lbm/ sack of cement

# 5) Additives for Controlling Lost Circulation

Lost circulation is define as the loss to induced fractures of either whole drilling fluid or cement slurry used in drilling or completing the well. It should not be confused with the volume decrease resulting from filtration or the volume required filling new hole.

Lost Circulation Control Additive	Amount Used ( wt% of Cement)		
Gilsonite	5 to 50 lbm/sack		
Perlite	0.5 to 1 cu ft/ sack		
Walnute Shells	1 to 5 lbm/ sack		
Coal	1 to 10 lbm/ sack		
Cellophane	0.125 to 2 lbm/sack		
Nylon	0.125 to 0.25 lbm/ sack		

Table 6. Common Lost Circulation Control Additive in Cement Slurries<sup>[2]</sup>

# 6) Filtration Control Agent

The filter loss of cement slurries is lowered with additives to prevent premature dehydration or loss of water against porous zones, protect sensitive formation and improve squeeze cementing. Two most widely used filtration control material are organic polymer and friction reducers <sup>[2]</sup>.

Table 7. Common Filtration Control Agent in Cement Slurries<sup>[2]</sup>

Filtration Control Agent	Amount Used ( wt% of Cement)
Cellulose	0.5 to 1.5
Dispersant	0.5 to 1.25
Carboxymethyl hydroxyethyl cellulose	0.3 to 1.0

Latex additives	1.0 gal/ sack
Nylon	0.125 to 0.25 lbm/ sack

### 7) Friction Reducer

Friction reducer agents are added into cement slurries to improve the cement slurries flow properties. Dispersed slurries will have lower viscosity and can be pumped in turbulence at lower pressure thereby minimizing the horsepower required and lessening the chances of lost circulation and premature dehydration <sup>[2]</sup>.

Friction Reducer Agent	Amount Used ( ldm/sack of Cement)		
Polymer			
-Blend	0.3 to 0.5		
-Long Chain	0.5 to 1.5		
Sodium Chloride	1 to 16		
Calcium Lignosulfonate, organic acid	0.5 to 1.5		

## 2.2 Design Calculation

Prior to the cement slurry design, studies had been taken in order to determine the concentration of additive and other material into slurry design. Equation 5 and Equation 6 will show the formula for calculation of weight and volume for silica fumes while Equation 7 and Equation 8 will show the formula for weight and volume of calculation chloride <sup>[2]</sup>.

$$Ws = \frac{X}{100} \times Wc.$$
 (5)

$$Vs = \frac{Ws}{\rho s} \tag{6}$$

$$Wca = \frac{Y}{100} \times Ww....(7)$$

$$Vca = \frac{Wca}{\rho ca}$$
(8)

Equation 9 and Equation 10 below will show the formula to calculate the weight for additive which will be added into slurry design.

$$Wa = \frac{B}{94} \times Wc \times \gamma \times 8.33.$$

$$Va = \frac{wa}{\rho a} \tag{10}$$

While the formula for water weight and volume were shown in Equation 11 and Equation 12.

$$Ww = \frac{c}{100} \times Wc.$$
 (11)

$$Vw = \frac{Ww}{\rho w}$$
(12)

Several physical properties of cement slurry can be calculated such as total weight, total volume and density as shown in Equation 13, 14 and 15.

$$Wt = Ws + Wca + Wa + Ww.$$
<sup>(13)</sup>

$$Vt = Vs + Vca + Va + Vw$$

$$\rho t = \frac{Wt}{Vt}.$$
(15)

### 2.3 Literature Review

Squeeze cementing is the process of applying hydraulic pressure to force or squeeze cement slurry into a formation void or against a porous zone <sup>[2]</sup>. Whether a cement squeeze operation result in annular seal depend heavily on how far the cement can penetrate and disperse in the fine channel of the partially cemented annulus. In most cases the conventional cement or microfine cement slurry will dehydrate and bridge off before it can achieve its objectives <sup>[10]</sup>. During the squeeze cement operation the cement slurry is subjected to differential pressure against the permeable formation. This process occurs in a cement squeeze operation regardless of the method used and occurs to a lesser extent when a circulation squeeze is performed <sup>[11]</sup>. The result from this process is filtration, filter cake deposition and fracturing of formation.

When squeeze against a formation of given permeability the rate at which slurry dehydration decreases is directly related to the fluid loss rate <sup>[12]</sup>. This show that during squeeze against high permeable formation, a slurry with high fluid loss rate dehydrate rapidly which may resulting the wellbore choked by filter cake and channel that suppose to accept cement would bridge off<sup>[1]</sup>. The requirement fluid loss rate for squeeze cementing is 50-200cc/30min<sup>[13]</sup>.

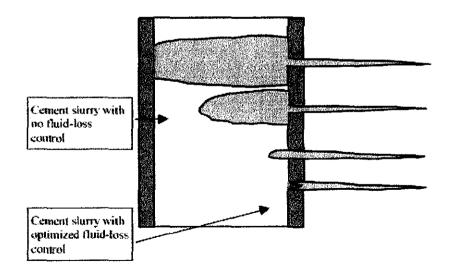


Figure 4. Filter Cake Deposition<sup>[1]</sup>.

Previous studies which had been done by Tsivilis and Parissakis showed that cement fineness mainly affects strength at early age (before 7 days) while chemical and mineralogical parameters influence strength at a later stage. They have also presented three laws to determine compressive strength at three ages (2, 7 and 28 days) in relation with the clinker compounds and/or the fineness parameter <sup>[14]</sup>.

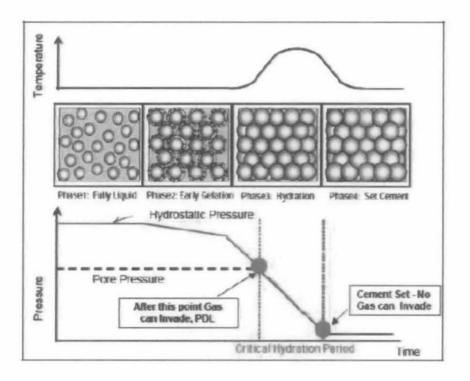


Figure 5. Different Phase in Setting of Cement Slurry<sup>[15]</sup>.

Several factors will play major role in the phase of slurry design such as temperature and pressure, type of cement, filtration control, quantity of cement, workover fluids, wellhead equipment, hole condition, cement strength and final squeeze pressure <sup>[2]</sup>. Successful placement of cement slurry for squeeze cementing usually requires slurry possessing excellent fluid loss control to prevent premature slurry dehydration, low viscosity for ease of entry into the channel, retarder if longer placement timers or greater bottom hole temperature are expected and compressive strength comparable to its primary cement originally placed<sup>[16]</sup>. This show that cement slurry design would be a really complicated studies which encompass all the affecting factors.

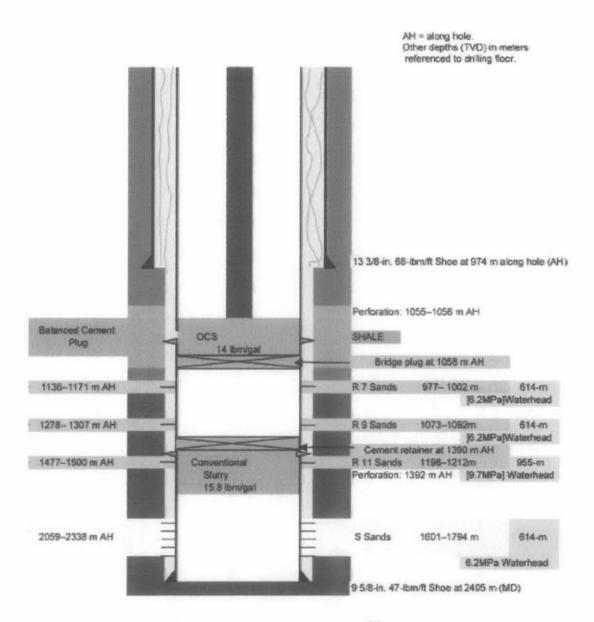


Figure 6. The Duyong B-4 Well Schematic<sup>[1]</sup>.

In order to overcome risks associated with squeeze cementing operation, one must first understand and create the optimized cement slurry design. A research had been done before which able to create innovative cement system which had compressive strength of 22MPa<sup>[1]</sup>. Therefore, this project was not impossible and it has been able to design optimized cement slurry design for squeeze cementing solution which will provide annular isolation in Duyong field, Malaysia.

# CHAPTER 3 METHODOLOGY

## 3.1 Research Methodology

Intensive investigation has been conducted to ensure that research went on smoothly. At first, the study on conventional cement slurry for squeeze cementing was been conducted to identify all the parameters such as fluid loss and compressive strength. Then a details study was covered on the factors affecting filtration control and also compressive strength of the set cement. After that, identifying the current additive of widely used in cement design will be done in order to determine the specification of the composition. Next, research on the cement slurry design has been continued by conducting several experiment activities. The outcome from the experiment will be used to create the specific cement slurry design for Duyong field as for the squeeze cementing operation.

## **3.2 Flow Chart**

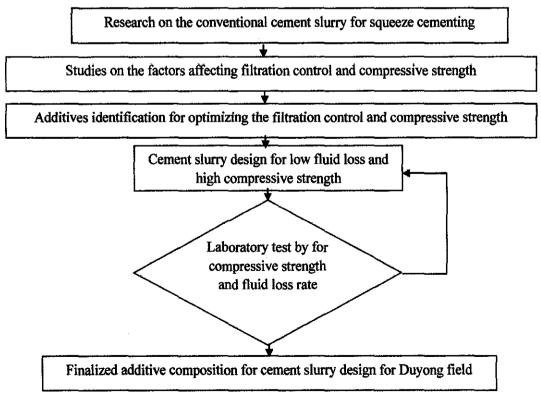


Figure 7. Flow Chart of the Research

# 3.3 Gantt Chart and Key Milestone

	2011										
		FYP 1						FYP 2			
Activities	J	F	М	A	м	L	J	Α	S		
Project planning and literature review											
Processing conventional cement slurry											
Measurement of filtration control and compressive strength of conventional cement slurry											
Studies on the factors affecting filtration control in cement slurry (obj. 1)											
Studies on the factors affecting set cement compressive strength ( obj. 2)											
Identifying additives affecting filtration control and compressive strength											
Designing optimize cement slurry composition for Duyong field (obj. 3)											
Measurement of physical properties of optimize cement slurry											
Research documentation						1					

					2011				
	FYP 1					FYP 2			
Milestone	J	F	Μ	Α	м	J	Ĺ	Α	S
Completion of conventional cement slurry composition and formulation									
Completion of optimizing filtration control		Γ							
Completion of optimizing set cement compressive strength									
Completion of designing optimized cement slurry composition for Duyong field									
Completion of filtration control and set cement compressive strength properties for optimize cement slurry									
Project completion									

## **3.4 Project Activities**

Table 10. Project Activities through the Final Year Project

Activities	From Date	To Date
Processing conventional cement slurry	1/03/2011	1/04/2011
Measurement of filtration control and compressive strength of conventional cement slurry	1/03/2011	1/04/2011
Studies on the factors affecting filtration control in cement slurry (obj. 1)	1/03/2011	1/05/2011
Studies on the factors affecting set cement compressive strength (obj. 2)	1/04/2011	1/05/2011
Identifying additives affecting filtration control and compressive strength	1/05/2011	1/06/2011
Designing optimize cement slurry composition for Duyong field (obj. 3)	1/05/2011	1/07/2011
Measurement of physical properties of optimize cement slurry	1/06/2011	1/08/2011
Research documentation	1/08/2011	1/09/2011

## **3.5 Material Selection**

In this project, reference books and research paper were the essential source of data. Most of the books and research paper were available at the university's library. After the research and studies, several materials had been identified to be used in this project. Most of the material was supplied by BJ Services Company due to economical reason and also availability of product.

## 3.5.1 Portland Type G Cement

This type of cement is derived by grinding Portland cement clinker with one or more types of calcium sulfates with Portland cement clinker. No additives is added during production of Type G oil well cement except clinker and calcium sulfate The aimed for this type of cement is to be used in oil well cementing operation. Type G oil well cement has high sulfate-resistant (HSR) and moderate sulfate-resistant (MSR) grades <sup>[2]</sup>.

Percentage (%)
50
30
5
12

Table 11: Composition of Oil Well Type G Cement<sup>[2]</sup>.

## 3.5.2 Silica Fume

Silica fume is a byproduct of producing silicon metal or ferrosilicon alloys. It is really beneficial to be used in cement slurry composition. As of its chemical and physical properties, it is a very reactive pozzolan. Cement slurry which containing silica fume can have very high strength and can be very durable. In this project the silica fume used was obtained from Elkem materials in dry densified form.

Table	12: XRF	of Silica	Fume <sup>[17]</sup> .
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Oxide	Percentage (%)
Content	
SiO <sub>2</sub>	96.36
Al <sub>2</sub> O <sub>3</sub>	0.21
Fe <sub>2</sub> O <sub>3</sub>	0.77
CaO	0.24
MgO	0.52

SO <sub>3</sub>	0.55
K₂O	1.02
Na <sub>2</sub> O	0.12

## 3.5.3 Accelerator (Calcium Chloride)

Calcium Chloride was the most effective of all cement accelerators. It compass of the salt of calcium and chlorine. It is a very hygroscopic material and need to be store in tightly-sealed air-tight containers <sup>[18]</sup>.

## 3.5.4 Fluid Loss Additive (FL-45LS)

FL-45LS was a liquid additive which was function to control the filtrate loss of the cement slurry for bottom hole condition. This additive basically was an anionic blend of high molecular weight synthetic copolymer and surfactant. The mechanism of controlling fluid loss is by absorption and conformation of macro molecules at the solid/solution interface. It means that, it will attach to the cement particles and thicken the interstitial water. This additive can be used in either sea water or fresh water but react best in light brines (up to 5% salt BWOW)<sup>[19]</sup>.

Appearance	Clear, colorless
рН	4-5.5
Specific Gravity	1.04
Boiling Point	>100 °C
Ionic Change`	Anionic

Table 13: Properties of FL-45LS<sup>[19]</sup>.

### 3.5.5 Dispersant (CD-31LS)

CD-31LS was the additive that in liquid form. It was added into mixing water to improve rheological properties of cement slurries. It physical property was dark brown liquid. Basically it was a highly polymerized naphthalene sulfonate. It will react effectively when using alongside with the FL-45LS <sup>[19]</sup>.

Appearance	Dark brown liquid, slight odor
рН	9-10
Specific Gravity	1.16
Boiling Point	212 °F
Solubility in water	Miscible

Table 14:Properties of CD-31LS<sup>[19]</sup>.

#### 3.5.6 Fluid Loss Additive (BA-86L)

BA-86L was a styrene-butadiene latex cement additive. It provides excellent fluid loss control, low viscosity, enhanced bonding and acid resistance. This will improve the fluid loss performance of the cement slurry <sup>[19]</sup>.

Table 15: Properties of BA-86L<sup>[19]</sup>.

Appearance	Milky white liquid, slight fishy odor	
Density	8.4 ppg	
Specific Gravity	1.007	
Absolute Volume	0.11905 gal/lb	
Solubility in water	Miscible	

## 3.5.7 Fresh Water

The fresh water is the normal tap water which is abundantly available at the laboratory.

Table 16: Properties of Fresh Water<sup>[2]</sup>.

Appearance	Colourless	
Density	8.3 ppg	
Specific Gravity	1	
Boiling Point	100°C	

## **3.6 Machinery**

Laboratory equipment also has been required for testing and properties evaluation. Two test were conducted which were compressive strength test and filtration control test. The equipment already available in Cement Laboratory under Geoscience and Petroleum Engineering Department, University Technology PETRONAS.

## 3.6.1 Mixing Device

The purpose of this machine is to blend and mix the liquid and solid component of the cement to create the cement slurry. This device has already following the specification agreed by the API Standard <sup>[6]</sup>. Laboratory manual had stated the standard operating procedure for the device <sup>[20]</sup>.



Figure 8. The Mixing Device

- a) Pour appropriate amount of water into the mixer container
- b) Turn the power switch to ON position
- c) Press the MIX 1 switches until it clicks into position
- d) Place the FIXED/VARIABLE switch to the VARIABLE position
- Press the START/RESET push button to start the motor and begin the timer countdown from 90 second.
- f) Add the cement to the water during the first 15 seconds while mixing at low speed (typically 4000rpm)
- g) After the cement has been added, place the cover on the mixer container.
- h) When the timer reach 35 seconds, press the MIX 2 button and mix on high speed (typically 12000 rpm) for 35 seconds. When the timer reach zero, the motor will stop automatically.

## 3.6.2 Curing Chamber



Figure 9. The Machine of HPHT Cement Curing Chamber

The objective of this equipment is to allow the cement to be cured in desired pressure and temperature. The standard operating procedure has been described in laboratory manual <sup>[20]</sup>:

- a) Close all valve and turn the unit on.
- b) Program the temperature ramp and soak parameters into temperature controller.
- c) Assemble each pair of mold bodies and fill with cement slurry according with API Specification 10.
- d) Lower the bucket full of molds into the pressure vessel.
- e) Make certain the cylinder plug threads are thoroughly lubricated and tighten securely by hand. Tighten the set screw in the plug.
- f) Insert thermocouple into the opening in the center of cylinder plug.
- g) Fill the pressure vessel with oil by opening Air Supply valve.
- h) Adjust the pressure in the vessel as desired for the start of the test
- i) Turn heater switch to the on position.
- j) Turn the timer switch to the on position

## 3.6.3 Compressive Strength Tester



Figure 10. The Machine of Compressive Strength Tester

The objective of this equipment is to measure the compressive strength of the set cement.

- a) Turn the unit on.
- b) Place the cement specimen in the lower platen of the hydraulic cylinder.
- c) Adjust the upper platen so that is touching the specimen.
- d) Open the compressive strength tester software on the PC
- e) From the 'Edit' menu select 'Option'.
- f) In the 'Data File Directory' choose the folder you would like the test data to be saved in.
- g) On the main screen, input the height of specimen (in inches) into the 'Cube Height' field.
- h) From the 'Edit' menu, select the 'File Data'
- i) Fill in all of the relevant information and click 'Ok'. This information is for display only and will not affect the test.
- j) Back on the main screen, choose a loading rate from the drop down menu.
  - 4000 psi/min- Use this setting if you expect the specimen to break more than 500 psi
  - 1000psi/min-Use this setting if you expect the specimen to break at less than 500psi
  - Auto- Set the load rate at 1000psi/min until it reaches 500psi, then increase the load rate to 4000psi/min for the rest of the test.
- k) Click the 'Pump On' button to start the pump. Fluid will now be circulating throughout the system, but the hydraulic ram will not yet be moving.
- Click and hold the 'Run Test' button to begin the test. The hydraulic ram will begin applying pressure to the specimen.
- m) Hold down 'Run Test' button while observing the specimen. When the specimen fails release the 'Run Test' button to stop the test and the pump. The software will then ask if you would like to print the results of the test.
- n) The 'Max Load (psi)' field show that maximum load that can be applied to the specimen before the test ended. This value is the compressive strength of the specimen.

o) When done, close the software by selecting 'Exit' from the 'File' menu. Do
not close the software by clicking the X in the upper right hand corner of the
screen.



#### 3.6.4 High Pressure and High Temperature Filter Press

Figure 11. The Equipment for Filtration Control

The objective of this experiment is to measure the fluid loss of the cement slurry. The procedure for filtration rate at 1000psi & 170°F experiment has been available in laboratory manual <sup>[20]</sup>.

- a) Detach the mud cell from the filter press frame
- b) Remove bottom of filter cell, place right size filter paper in the bottom of the cell.
- c) Introduce slurry to be tested into cup assembly, putting filter paper and screen on top of mud tighten screw clamp.
- d) With the air pressure valve closed, clamp the mud cup assembly to the frame while holding the filtrate outlet end finger tight.
- e) Place a graduated cylinder underneath to collect filtrate.
- f) Open air pressure valve and start timing at the same time.
- g) Report cc of filtrate collected for specified intervals up to 30 minutes.
- h) Tabulate the result in an appropriate table

## CHAPTER 4 RESULTS & FINDINGS

#### 4.1 Expected Result

This research emphasizes in the filtration control and set cement compressive strength. The expected outcome from this research is to create cement slurry design with the compressive strength more than 22MPa (3190psi) and low fluid loss (<15cm<sup>3</sup>/30 min).

As for the filtration control, there will be several factors which contribute to the result such as additive used, temperature, pressure and permeability. To measure filtration characteristics of cement slurries, the API specifies a standardized 30-minutes test at 100 psi or 1000 psi <sup>[6]</sup>.

As for the compressive strength, the following factor will high affect the results which are additive used, thickening time, pressure and temperature.

	Table 17. The Effect of Additi	ve (CD-31L) in Com	pressive Strength <sup>[19]</sup>
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COMPRESSIVE STRENGTH DEVELOPMENT CLASS "G" CEMENT WITH CD-31L (IN psi)								
CD-31L	Density	24 HRS	24 HRS					
(ghs)	ppg	(at 170°F)	(at 200°F)					
0.1	17	5578	5888					
0.14	17.5	6975	7075					

#### 4.2 Slurry Design

Experiment and studies had been conducted in order to come out with several slurry designs. Parameters of compressive strength and filtration control had been emphasizes throughout the design process.

Cement	Type G	Silica	CACL	FL-45LS	CD-	BA-86L	Water
Sampl <del>e</del>	Cement (%)	Fume (% BWOC)	(%BWOW)	(gps)	31LS (gps)	(gps)	(%)
Α	100	-	-	-	•	-	44
В	100		-	0.45	0.9	-	44
С	100	-	2	0.45	0.9	0.5	44
D	100	10	2	0.45	0.9	-	45
E	100	-		0.5	1	-	44
F	100	-	3	0.5	1	-	44
G	100	10	3	0.5	1		45
Н	100	10	3	0.5	1	0.5	45

Table 18 . Cement Slurry Design

## 4.3 Experimental

Experiment had been conducted in order to evaluate the outcome properties of each slurry design according to the reference provided in the laboratory <sup>[21]</sup>. Prior to the experiment the value for each material had been calculated and tabulated in the Microsoft Excel.

### 4.3.1 Sample A

This was the base sample as using the conventional cement slurry without any other additives added into the design. The data has been used as base line to compare with other sample. Table 19 was showing the composition of the conventional cement slurry.

MATERIALS	% OF MAT. Or GPS	S.G	WT. OF MAT. (Ibs)	VOLUME (gal)	WEIGHT (gm)	VOLUME (cc)
Cement Type G	100	3.14	0.88	0.03	400.00	127.39
Calcium Chloride	0	2.15	0.00	0.00	0.00	0.00
Silica Fumes	0	2.65	0.00	0.00	0.00	0.00
FL-45LS	0	1.04	0.00	0.00	0.00	0.00
CD-31LS	0	1.16	0.00	0.00	0.00	0.00
BA-86L	0	1.01	0.00	0.00	0.00	0.00
Water	44	1.00	0.39	0.05	176.00	176.00
	<u> </u>	Total	1.27	0.08	576.00	303.39

Table 19. The Amount of Material Used for Mixing Sample A

This composition had created the cement slurry with the density of 15.8ppg and the volume of 304ml. This slurry has been put in the High Pressure and High Temperature apparatus in order to evaluate the filtration parameter. The condition for filtration was set constant throughout all samples which were at 170°F and 1000 psi. As this was the base case, it has show really bad filtration control because total burst had happened within the first 5 minutes. The experiment had been conducted three times in order to give the best value and by utilizing the formula the fluid loss rate obtain for this base case is 1017.26 ml and this slurry composition was not qualified for the squeeze cementing operation.

Time (min)	Raw 1 Filtrate (ml or g)	API Calculated ( if "blown out") 1 Filtrate (ml or g)	Raw 2 Filtrate (ml or g)	API Calculated ( if "blown out") 2 Filtrate (ml or g)	Raw 3 Filtrate (ml or g)	API Calculated ( if "blown out") 3 Filtrate (ml or g)	Average
0	0. 8/	0.00	0.6/	0.00	0.8/	0.00	0.00
2	130	1006.93	126	975.95	138	1068.90	1017.26
10	130	1006.93	126	975.95	138	1068.90	1017.26
15	130	1006.93	126	975.95	138	1068.90	1017.26
20	130	1006.93	126	975.95	138	1068.90	1017.26
25	130	1006.93	126	975.95	138	1068.90	1017.26
30	130	1006.93	126	975.95	138	1068.90	1017.26

Table 20. The Data of Filtration Loss for Sample A

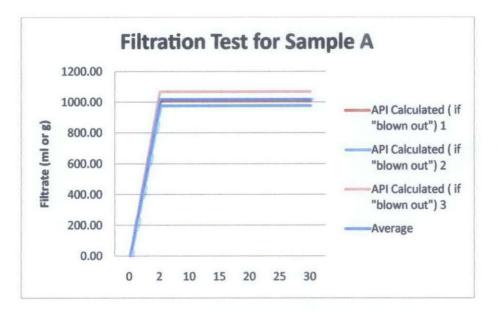


Figure 12. The Performance of Filtration Test for Sample A

As for the compressive strength, the base case had shown good performance under the specific bottom hole condition. As the design was for Duyong field, the cement was cured under temperature of 170°F and pressure of 1000 psi for 24 hours.

Strength	Pound	MPa	psi
Base Case 1	47.8	19.12	2773.12
Base Case 2	48.2	19.28	2796.33
Base Case 3	48.9	19.56	2836.94
Base Case 4	47.4	18.96	2749.92
Average	48.075	19.23	2789.08

Table 21. The Result of Compressive Strength for Sample A

The compressive strength from the base case had almost reached the objective target which was 22MPa. This had conclude that the conventional base cement slurry already had good performance on compressive strength however lot of adjustment need to be done on the filtration loss and the amount of filtrate produced was not acceptable for squeeze cementing operation.

#### 4.3.2 Sample B

This was the first sample that will be utilizing the additive. This slurry composition was encompassed of fluid loss additive (FL-45LS) and dispersant (CD-31LS). Table 22 was the amount that has been used in the studies.

MATERIALS	% OF MAT. Or GPS	S.G	WT. OF MAT. (lbs)	VOLUME (gal)	WEIGHT (gm)	VOLUME (cc)
Cement Type G	100	3.14	0.88	0.03	400.00	127.39
Calcium Chloride		2.15	0.00	0.00	0.00	0.00
Silica Fumes		2.65	0.00	0.00	0.00	0.00
FL-45LS	0.45	1.04	0.04	0.00	16.59	15.95
CD-31LS	0.9	1.16	0.08	0.01	37.01	31.90
BA-86L		1.01	0.00	0.00	0.00	0.00
Water	44	1.00	0.39	0.05	159.41	159.41
		Total	1.39	0.09	613.01	281.06

Table 22. The Amount of Material Used for Mixing Sample B

This amount of material will generate the cement slurry with density of 16.1 ppg and volume of 280ml. The data obtained from this had shown good result on the fluid loss or filtration control. Table 23 had detailed on the result.

	Raw 1	API Calculated 1	Raw 2	API Calculated 2	Raw 3	API Calculated 3	Average
Time (min)	Filtrate (ml or g)	Filtrate (ml or g)	Filtrate (ml or g)	Filtrate (mi or g)	Filtrate (ml or g)	Filtrate (ml or g)	
0	0.0	0.0	0.0	0.0	0.0	0.0	0.00
5	1.0	2.0	0.9	1.8	1.2	2.4	2.07
10	1.6	3.2	1.4	2.8	1.6	3.2	3.07
15	2.0	4.0	2.2	4.4	2.4	4.8	4.40
20	2.6	5.2	3.0	6.0	3.2	6.4	5.87
25	3.4	6.8	3.8	7.6	4.0	8.0	7.47
30	5.0	10.0	4.6	9.2	5.6	11.2	10.13

Table 23. The Data of Filtration Loss for Sample B

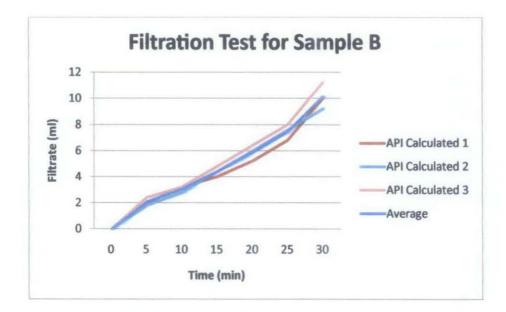


Figure13. The Performance of Filtration Test for Sample B

As usual the experiment was repeated three times in order to get precise data. Sample B had shown good performance in filtration control and meet the criteria for squeeze cementing.

The cement had been cured for 24 hours before being test in Compressive Strength Tester. Table 24 was the data obtained from test.

Strength	Load (kN)	MPa	Psi
Sample B 1	58.9	23.56	3417.09
Sample B 2	60.6	24.24	3515.71
Sample B 3	49.8	19.92	2889.15
Sample B 4	55.9	22.36	3243.04
Average	56.3	22.52	3266.25

Table 24. The Result for Compressive Strength for Sample B

The compressive strength data had shown decreasing from the conventional cement as the influence of fluid loss additive. This had concludes that the slurry composition already had good performance on the filtration control and also compressive strength.

## 4.3.3 Sample C

Sample C had been improved with the addition of others additive which were calcium chloride (CACL), latex (BA-86L), fluid loss additive (FL-45LS) and dispersant (CD31-LS). Table 25 showed the amount and composition of this slurry.

MATERIALS	% OF MAT. Or GPS	S.G	WT. OF MAT. (lbs)	VOLUME (gal)	WEIGHT (gm)	VOLUME (cc)
Cement Type G	100	3.14	0.88	0.03	400.00	127.39
Calcium Chloride	2	2.15	0.00	0.00	2.09	0.97
Silica Fumes		2.65	0.00	0.00	0.00	0.00
FL-45LS	0.45	1.04	0.04	0.00	16.59	15.95
CD-31LS	0.9	1.16	0.08	0.01	37.01	31.90
BA-86L	0.5	1.01	0.04	0.00	17.90	17.72
Water	44	1.00	0.23	0.03	104.50	104.50
		Total	1.27	0.08	578.09	298.44

Table 25. The Amount of Material Used for Mixing Sample C

This amount had created cement slurry with the density of 16.1 ppg and volume of 298ml. The slurry then undergone filtration test at the bottom hole condition. Table 26 was the data obtained from the filtration test.

API API API Calculated Calculated Calculated Raw 1 1 Raw 2 2 Raw 3 3 Average Time Filtrate Filtrate Filtrate Filtrate Filtrate Filtrate (min) (ml or g) (ml or g) (mi or g) (ml or g) (ml or g) (ml or g) 0.0 0.00 0 0.0 0.0 0.0 0.0 0.0 5 4.4 3.0 5.20 2.2 6.0 2.6 5.2 10 2.8 5.6 3.2 6.4 3.2 6.4 6.13 7.2 7.87 15 3.6 4.0 8.0 4.2 8.4 20 5.2 10.4 4.8 9.6 9.87 4.8 9.6 25 5.4 10.8 5.8 11.6 6.2 12.4 11.60 30 7.0 14.0 6.6 13.2 7.8 15.6 14.27

 Table 26. The Data of Filtration Loss for Sample C

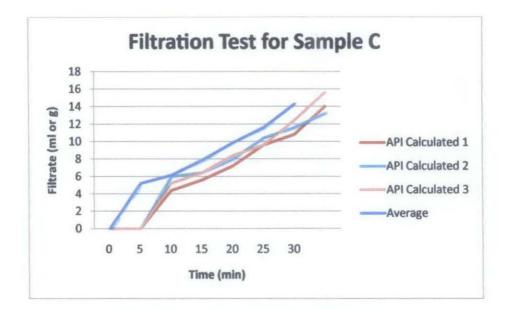


Figure 14. The Performance of Filtration Test for Sample C

Experiment was repeated three times in order to compare the data obtained. Sample C had shown moderate performance on the filtration control.

Sample C had been cured for 24 hours in the same bottom hole condition which temperature of 170°F and pressure of 1000 psi. The result for the test had been shown in table 27.

Strength	Pound	Mpa	psi
Sample 1	39.8	15.92	2309.00
Sample 2	32.7	13.08	1897.09
Sample 3	34.9	13.96	2024.73
Sample 4	36.9	14.76	2140.76
Average	36.1	14.43	2092.89

Table 27. The Result for Compressive Strength for Sample C

This can conclude that by adding latex additive, it had reduced the compressive strength of the set cement. Sample C had show moderate performance on fluid loss or filtration control and low on compressive strength of the cement

#### 4.3.4 Sample D

Sample D had been improved with the addition of other silica fumes which had been generally know for increasing the compressive strength. The amount and composition of this slurry is as in table 28.

MATERIALS	% OF MAT. Or GPS	S.G	WT. OF MAT. (lbs)	VOLUME (gal)	WEIGHT (gm)	VOLUME (cc)
Cement Type G	100	3.14	0.88	0.03	400.00	127.39
Calcium Chloride	2	2.15	0.01	0.00	2.53	1.18
Silica Fumes	10	2.65	0.09	0.00	40.00	15.09
FL-45LS	0.45	1.04	0.04	0.00	16.59	15.95
CD-31LS	0.9	1.16	0.08	0.01	37.01	31.90
BA-86L		1.01	0.04	0.00	0.00	0.00
Water	45	1.00	0.23	0.03	109.82	109.82
····· ··· ··· ··· ··· ··· ··· ··· ···		Total	1.37	0.08	605.94	301.33

Table 28. The Amount of Material Used for Mixing Sample D

This amount used had created cement slurry with the density of 16.3 ppg and volume of 301ml. Table 29 shows the data of the filtration test.

	Raw 1	API Calculated 1	Raw 2	API Calculated 2	Raw 3	API Calculated 3	Average
Time (min)	Filtrate (ml or g)	Filtrate (ml or g)	Filtrate (ml or g)	Filtrate (mi or g)	Filtrate (ml or g)	Filtrate (ml or g)	
0	0	0.0	0.0	0.0	0.0	0.0	0.00
5	0.6	1.2	1.0	2.0	1.2	2.4	1.87
10	2.0	4.0	2.6	5.2	2.8	5.6	4.93
15	2.6	5.2	3.4	6.8	3.0	6.0	6.00
20	3.2	6.4	3.8	7.6	3.4	6.8	6.93
25	3.6	7.2	4.0	8.0	3.6	7.2	7.47
30	4.0	8.0	4.2	8.4	3.8	7.6	8.00

Table 29. The Data of Filtration Loss for Sample D

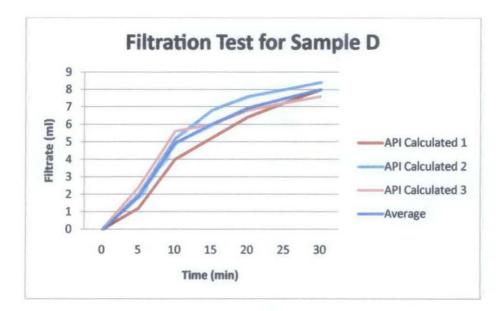


Figure 15. The Performance of Filtration Test for Sample D

Experiment is repeated three times in order to compare the data obtained. Sample D had shown good performance on the filtration control. Sample D had been cured for 24 hours in same bottom hole condition. The result for the test had been tabulated in table 30.

Table 30.	The Result for	Compressive	Strength for	Sample D

Strength	Pound	Мра	psi
Sample 1	62.70	25.08	3637.55
Sample 2	53.80	21.52	3121.21
Sample 3	64.50	25.80	3741.97
Sample 4	57.90	23.16	3359.07
Average	59.73	23.89	3464.95

Based on the result of compressive strength test, it had showed that silica fumes really increase the compressive strength. This had been proved when compared with the sample B whereby the compressive strength just only 22MPa. Sample D already meet the requirement for Duyong's field Malaysia.

#### 4.3.5 Sample E

Sample E had been improved with the increment of concentration on fluid loss additive and dispersant. The amount and composition of this slurry was as tabulated in table 31.

MATERIALS	% OF MAT. Or GPS	S.G	WT. OF MAT. (lbs)	VOLUME (gai)	WEIGHT (gm)	VOLUME (cc)
Cement Type G	100	3.14	0.88	0.03	400.00	127.39
Calcium Chloride		2.15	0.00	0.00	0.00	0.00
Silica Fumes		2.65	0.00	0.00	0.00	0.00
FL-45LS	0.5	1.04	0.04	0.00	18.43	17.72
CD-31LS	1	1.16	0.09	0.01	41.12	35.45
BA-86L		1.01	0.04	0.00	0.00	0.00
Water	44	1.00	0.26	0.03	116.45	116.45
		Total	1.27	0.08	576.00	297.01

Table 31. The Amount of Material Used for Mixing Sample E

This amount used will create cement slurry with the density of 16.2 ppg and volume of 297ml. Table 32 was showing the data for the filtration test.

	Raw 1	API Calculated 1	Raw 2	API Calculated 2	Raw 3	API Calculated 3	Average
Time	Filtrate	Filtrate	Filtrate	Filtrate	Filtrate	Filtrate	
(min)	(ml or g)	(ml or g)	(ml or g)	(ml or g)	(ml or g)	(ml or g)	
0	0	0	0	0	0	0	0.00
5	0.6	1.2	0.6	1.2	1.0	2.0	1.47
10	1.2	2.4	1.6	3.2	1.8	3.6	3.07
15	2.6	5.2	2.4	4.8	3.2	6.4	5.47
20	2.8	5.6	3.0	6.0	3.8	7.6	6.4
25	3.2	6.4	3.6	7.2	4.6	9.2	7.6
30	4.4	8.8	4.8	9.6	5.8	11.6	10.0

Table 32. The Data of Filtration Loss for Sample E

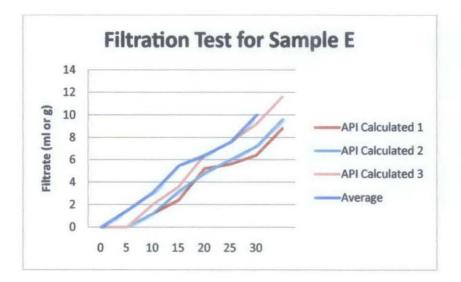


Figure 16. The Performance of Filtration Test for Sample E

In order to obtained precise data the experiment was repeated three times. Sample E had been cured for 24 hours in same bottom hole condition. The result for the test had been tabulated in table 33.

Table 33. The Result for G	Compressive	Strength for	Sample E
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Strength	Pound	Mpa	psi
Sample 1	54.10	21.64	3138.62
Sample 2	60.60	24.24	3515.71
Sample 3	55.60	22.24	3225.64
Sample 4	59.20	23.68	3434.49
Average	57.38	22.95	3328.62

Sample E had show good performance on both fluid loss or filtration control and compressive strength of the cement. This can be found that the increment for fluid loss additive had improved the filtration control.

#### 4.3.6 Sample F

Sample F had been improved with the addition of accelerator into the slurry design. The amount and composition of this slurry is as tabulated in table 34.

MATERIALS	% OF MAT. Or GPS	S.G	WT. OF MAT. (lbs)	VOLUME (gal)	WEIGHT (gm)	VOLUME (cc)
Cement Type G	100	3.14	0.88	0.03	400.00	127.39
Calcium Chloride	3	2.15	0.01	0.00	3.49	1.62
Silica Fumes		2.65	0.00	0.00	0.00	0.00
FL-45LS	0.5	1.04	0.04	0.00	18.43	17.72
CD-31LS	1	1.16	0.09	0.01	41.12	35.45
BA-86L		1.01	0.04	0.00	0.00	0.00
Water	44	1.00	0.26	0.03	116.45	116.45
		Total	1.28	0.08	579.49	298.63

Table 34 . The Amount of Material Used for Mixing Sample F

This amount used had created cement slurry with the density of 16.2 ppg and volume of 298ml. Table 35 and figure 17 were showing the data for the filtration test.

	Raw 1	API Calculated 1	Raw 2	API Calculated 2	Raw 3	API Calculated 3	Average
Time (min)	Filtrate (ml or g)	Filtrate (ml or g)	Filtrate (ml or g)	Filtrate (ml or g)	Filtrate (ml or g)	Filtrate (ml or g)	
0	0	0	0	0	0	0	0.00
5	1.0	2.0	0.4	0.8	0.6	1.2	1.33
10	1.6	3.2	2.0	4.0	1.2	2.4	3.2
15	2.4	4.8	2.8	5.6	2.4	4.8	5.07
20	3.2	6.4	3.6	7.2	3.2	6.4	6.67
25	4.8	9.6	4.2	8.4	3.8	7.6	8.53
30	5.2	10.4	5.4	10.8	4.2	8.4	9.87

Table 35. The Data of Filtration Loss for Sample F

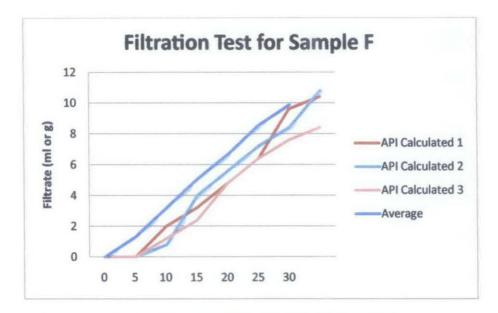


Figure 17. The Performance of Filtration Test for Sample F

Experiment was repeated three times in order to compare the data obtained. Sample F had been cured for 24 hours in same bottom hole condition. The result for the test had been tabulated in table 36.

Strength	Pound	Мра	psi
Sample 1	61.20	24.48	3550.52
Sample 2	58.70	23.48	3405.49
Sample 3	64.60	25.48	3747.78
Sample 4	60.20	24.08	3492.51
Average	61.18	24.47	3549.07

Table 36. The Result for Compressive Strength for Sample F

Sample F had show good performance on both fluid loss or filtration control and compressive strength of the cement and has properties better than sample D. This had shown that calcium chloride had improved the compressive strength of the set cement.

## 4.3.7 Sample G

Sample G had been improved with the addition of silica fumes into the slurry design. Table 37 had shown the amount and composition of this slurry.

MATERIALS	% OF MAT. Or GPS	S.G	WT. OF MAT. (lbs)	VOLUME (gal)	WEIGHT (gm)	VOLUME (cc)
Cement Type G	100	3.14	0.88	0.03	400.00	127.39
Calcium Chloride	3	2.15	0.01	0.00	3.61	1.68
Silica Fumes	10	2.65	0.09	0.00	40.00	15.09
FL-45LS	0.5	1.04	0.04	0.00	18.43	17.72
CD-31LS	1	1.16	0.09	0.01	41.12	35.45
BA-86L		1.01	0.00	0.00	0.00	0.00
Water	45	1.00	0.23	0.03	120.45	120.45
		Total	1.37	0.08	623.61	317.78

Table 37. The Amount of Material Used for Mixing Sample G

This amount used had created cement slurry with the density of 16.4 ppg and volume of 318ml. Table 38 and figure 18 had shown the data for filtration test.

	Raw 1	API Calculated 1	Raw 2	API Calculated 2	Raw 3	API Calculated 3	Average
Time (min)	Filtrate (ml or g)	Filtrate (ml or g)	Filtrate (ml or g)	Filtrate (ml or g)	Filtrate (ml or g)	Filtrate (ml or g)	
0		0	0	0	0	0	0.00
5	0.6	1.2	0.8	1.6	0.8	1.6	1.47
10	1.4	2.8	1.2	2.4	1.2	2.4	2.53
15	2.6	5.2	1.8	3.6	2.2	4.4	4.40
20	2.8	5.6	2.6	5.2	2.8	5.6	5.47
25	3.6	7.2	3.2	6.4	3.4	6.8	6.80
30	4.4	8.8	3.8	7.6	3.6	7.2	7.87

Table 38. The Data of Filtration Loss for Sample G

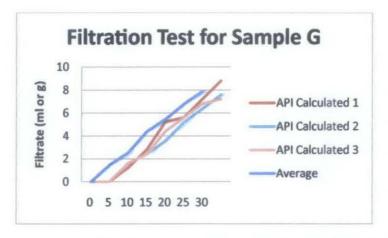


Figure 18. The Performance of Filtration Test for Sample G

Experiment was repeated three times in order to compare the data obtained. Sample G had been cured for 24 hours in bottom hole condition. The result for the compressive strength test had been tabulated in table 39.

Strength	Pound	Mpa	psi
Sample 1	64.60	25.84	3747.78
Sample 2	67.40	26.96	3910.22
Sample 3	62.80	25.12	3643.35
Sample 4	61.40	24.56	3562.13
Average	64.05	25.62	3715.87

Table 39. The Result for Compressive Strength for Sample G

Sample G had show best performance on both fluid loss or filtration control and compressive strength of the cement. This was the result of proper combination of additive and right concentration which had created the desired properties.

#### 4.3.8 Sample H

Sample H had been improved with the addition of latex into the slurry design. The amount and composition of this slurry is as in table 40.

MATERIALS	% OF MAT. Or GPS	S.G	WT. OF MAT. (lbs)	VOLUME (gal)	WEIGHT (gm)	VOLUME (cc)
Cement Type G	100	3.14	0.88	0.03	400.00	127.39
Calcium Chloride	3	2.15	0.01	0.00	3.08	1.43
Silica Fumes	10	2.65	0.09	0.00	40.00	15.09
FL-45LS	0.5	1.04	0.04	0.00	18.43	17.72
CD-31LS	1	1.16	0.09	0.01	41.12	35.45
BA-86L	0.5	1.01	0.04	0.00	17.90	17.72
Water	45	1.00	0.23	0.03	102.55	102.55
		Total	1.37	0.08	623.08	317.36

Table 40. The Amount of Material Used for Mixing Sample H

This amount used will create cement slurry with the density of 16.4 ppg and

volume of 318 ml. Table 41 was showing the data of the filtration test

Time (min)	Raw 1 Filtrate (ml or g)	API Calculated 1 Filtrate (ml or g)	Raw 2 Filtrate (ml or g)	API Calculated 2 Filtrate (ml or g)	Raw 3 Filtrate (ml or g)	API Calculated 3 Filtrate (ml or g)	Average
0	0	0	0	0	0	0	0.00
5	2.0	4.0	2.6	5.2	1.6	3.2	4.33
10	3.2	6.4	3.0	6.0	2.8	5.6	6.00
15	4.6	9.2	4.8	9.6	3.2	6.4	8.40
20	5.8	11.6	5.4	10.8	3.8	7.6	10.00
25	6.4	12.8	6.0	12.0	4.4	8.8	11.20
30	7.8	15.6	6.4	12.8	4.8	9.6	12.67

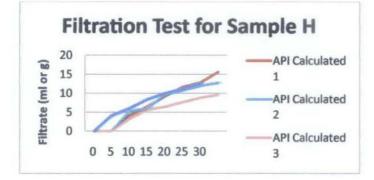


Figure 19. The Performance of Filtration Test for Sample H

Experiment was repeated three times in order to compare the data obtained. Sample H had been cured for 24 hours in bottom holes condition. The result for the test had been tabulated in table 42.

Strength	Pound	Мра	psi
Sample 1	52.70	21.08	3057.40
Sample 2	49.60	19.84	2877.55
Sample 3	52.00	20.80	3016.79
Sample 4	47.90	19.16	2778.92
Average	50.55	20.22	2932.66

Table 42. The Result for Compressive Strength for Sample H

Sample H had show low performance on both fluid loss or filtration control and compressive strength of the cement. This can be seen as the effect of latex additive in the slurry design.

#### 4.4 Analysis

The addition of additive into slurry composition had given really big changes in the cement properties. All the sample had been combine together and table 43 was showing the tabulated data.

Sample	API Calclulated 1	API Caiclulated 2	API Calciulated 3	Average (ml/30min)
Sample A	1006.93	975.95	1068.90	1017.26
Sample B	10.00	9.20	11.20	10.13
Sample C	14.00	13.20	15.60	14.27
Sample D	8.00	8.40	7.60	8.00
Sample E	8.80	9.60	11.60	10.00
Sample F	10.40	10.80	8.40	9.87
Sample G	8.8	7.6	7.2	7.87
Sample H	15.6	12.8	9.6	12.67

Table 43. The Result for Filtration Test

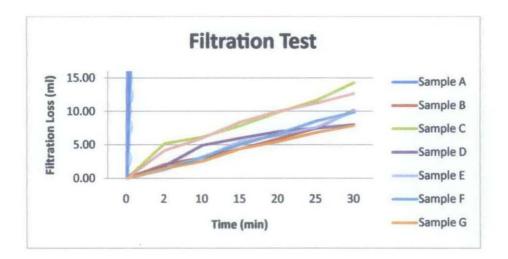


Figure 20. The Performance of Filtration Test for All Samples

Based on the result for all sample, sample A had shown the really bad filtration lost as this was the conventional cement slurry without any additive added into the slurry. The calculated fluid loss for this sample A is 1017.26 ml/30min. Sample G had shown really good performance on the filtration loss which was average 7.87 ml/30min. This result had met the requirement for Duyong's Field, Malaysia. This was because the addition of proper fluid loss additive, dispersant, silica fumes and accelerator. The ratio of 2:1 for fluid loss additive to dispersant had shown really good result in this project.

Sample	Pound	Mpa	psi
Sample A	48.08	19.23	2789.08
Sample B	56.30	22.52	3266.25
Sample C	36.08	14.43	2092.89
Sample D	59.73	23.89	3464.95
Sample E	57.38	22.95	3328.62
Sample F	61.18	24.47	3549.07
Sample G	64.05	25.62	3715.87
Sample H	50.55	20.22	2932.66

Table 44 . The Result for Compressive Strength

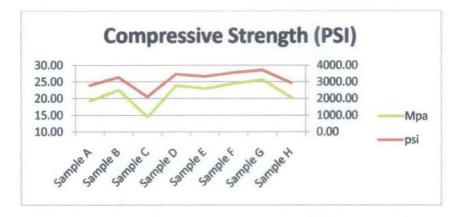




Table 44 and figure 21 had shown the combination of all sample for the compressive strength test. It can be seen that sample C had the lowest performance for compressive strength which average was about 14.43MPa. This can be concluded that the addition of latex into slurry design will decrease the compressive strength. As for the highest compressive strength for the entire sample is sample G with the compressive strength about 25.62MPa. This had cause by higher concentration of the dispersant which had densify the slurries resulting in stronger cement. The addition of silica fumes also improves the compressive strength.

As for Duyong's Field Malaysia, the suitable cement slurry design for this case was the sample G which had average fluid loss rate about 7.87ml/30min and the compressive strength of 25.62MPa. The cement slurry design would encompass of several elements which were silica fumes 10% (BWOC), calcium chloride 3%(BWOW), FL-45ls 0.5 gallon per sacks (gps), CD-31LS 1.0 gallon per sacks (gps), water 45% (BWOC).

## CHAPTER 5 CONCLUSION

Based on the result, this project had been successfully met the objective. Below was the conclusion for this project.

- The optimization of the filtration control had been achieved successfully by adding fluid loss additive together with dispersant. Data from experiment had shown that low filtrate loss had been achieved which was as low as 7.87ml/30min.
- 2) The compressive strength of the set cement had also been improved by adding the silica fumes, calcium chloride and dispersant into slurry design. Result from laboratory experiment had shown that the highest compressive strength was recorded at 25.62MPa which had been cured for 24 hours.
- As for the innovative design for squeeze cementing in Duyong's Field, Malaysia, data had shown that several additive would give the best performance such as low filtration loss of 7.87ml/30 min and high compressive strength of 25MPa. The design would encompass of type G cement, 10% of silica fumes, 3% calcium chloride, 0.5 gallon per sacks (gps) of fluid loss additive, 1.0 gallon per sacks (gps) of dispersant and water.

# CHAPTER 6 RECOMMENDATION

The project had been part of the step for squeeze cementing operation. Several suggestions on improving this studies and lead to successful squeeze cementing operation were listed below:

- 1) Always referred to the API standard so the result was comparable to the industry used.
- As for the machinery, always make sure the equipment had been properly serviced before the usage as to comply with the safety regulation and to make sure good result.
- 3) Prior to real squeeze cementing operation another details need to be consider in order to make sure the successful of the operation. Parameters such as thickening time, viscosity, density and lost circulation should be well understood and design accordingly to the desire depth.
- 4) Further studies would also be required prior to this squeeze cementing operation whereby the economical analysis could be performed to evaluate the cost of cementing materials, surface equipment, down holes equipment and others.
- 5) Details on job procedure also should be studies such as pumping pressure, injection rate, well clean up, well kick off and etc. All these will lead to the successful operation in Duyong's Field, Malaysia

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