New Blended Cement for Oil-Well Application

by

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Dissertation submitted in partial fulfillment of
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Bachelor of Engineering (Hons)
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CERTIFICATION OF APPROVAL

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Approved:

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SEPTEMBER 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.

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MOHAMAD AMIN ZIKRI BIN MOHAMAD FAUZI

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ABSTRACT

Blended cement by definition is cement consisting of an intimate and uniform blend of Portland cement and pozzolanic material. Blended cement is produced by intergrinding Portland cement with the other materials. For a long time, petroleum and cement industries were conducting investigations and field studies to improve compressive strength, thickening time, fluid loss and also microstructure. So, the objective of this project is to create new blended cement that improves compressive strength, thickening time, and fluid loss. With this new blended cement, a lot of improvements towards environment, society, petroleum industry and also in terms of financial benefits. Materials that used in this project were oil-well cement class G, silica fume and fly ash class C. Five samples of model have been made for experimenting in order to compare with the conventional cement. The compositions were starting with constant 50% for cement class G and 0% for silica fume and 50% fly ash class F. All these experiments will be conducted in the cement lab and supervised by lab technicians. After gaining the results, better composition that leads for improvements than conventional cement will be chosen based on compressive strength testing, thickening time and fluid loss. The compressive strength was tested with compression machine. Based on this research, blended cement will be produce using HPHT curing chamber, stirred fluid loss tester and consistometer. The results showed blended cement (sample 4) with composition of 50% cement class G, 37.5% silica fume and 12.5% fly ash class F results in higher compressive strength, better thickening time and fluid loss compared to other. In addition, blended cement is proven cheaper in term of cost compared to conventional cement since the materials are available abundantly. In conclusion, this project can provide new information to improve the quality of blended cement.

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LIST OF ABBREVIATION

API	American Petroleum Institute
FYP	Final Year Project
UTP	Universiti Teknologi PETRONAS
OPC	Ordinary Portland Cement
TT	Thickening Time
BWOW	By Weight of Water
HPHT	High Pressure High Temperature
SF	Silica Fume
FA	Fly Ash

CHAPTER 1
INTRODUCTION

1.1 Background of Study

Many advances have been made since the initial use of cement in the oil-producing industry to help isolate zones and to provide protection and support for the casing string. Essentially, these improvements can be categorized into two main parts which are mechanical and chemical components. Mechanical improvements have been made on blending and mixing equipment, tools, casing equipment and many other equipment/accessories while chemical improvements include many special additives and material for blending with cement to control fluid loss, thickening time, lost circulation and other properties indicted to be crucial for improved cementing jobs.

The first attempt to improve bonding was at the casing-cement and cement-formation interfaces done by Evans and Carter during early 1960's [1]. They were introducing to oil-industry of concepts such as hydraulic and shear bonding and other things such as condition of pipe surface needed for improved bonding. After that first attempts, a lot series of experiments and method to improved and make better bonding at the casing-cement and cement-formation interfaces. Latest finding was done by Wolsiefer [2] in 1991 which regarding utilization of silica fume in concrete admixture to improve bonding in cement. Due to high silica content, very fine particle size and extremely large surface area is a highly effective Pozzolanic material.

Another method to improve bonding is to add fly ash since its properties quite similar to silica fume. Based on one cement research group in Malaysia, in terms of fluid loss, thickening time, compressive strength and free water tested at atmospheric temperature and pressure and simulated reservoir condition, locally produces cement especially the pulverize fly ash cement proved to have the properties suitable for the application in the oil-well cementing operations [3].

Thus, the addition of another material in cement slurry seems to improve the properties and microstructure of the cement. However, further testing must be done in adding and mixing with additives to the cement and few others to exactly verify the justification.

1.2 Problem Statement

1.2.1 *Problem Identification*

For a long time, petroleum and cement industries were conducting investigations and field studies to improve compressive strength, thickening time and fluid loss of the cement including microstructure itself. Since basic oil-well cement has some problems such as gas leakage and migration, high permeability and also strength due to imperfection in its properties and microstructure, new blended cement have to make in order to improve cement properties and microstructure. Production of the cement also will pollute the environment by emitting carbon dioxide to the atmosphere.

1.2.2 *Significance of Project*

With this new blended cement, a lot of improvements towards environment, society, petroleum industry and also in terms of financial benefits. Production of Portland cement is highly gas pollutant which emits considerable amount of Carbon dioxide in the atmosphere. Carbon dioxide is well known for its ill-effects towards global warming as greenhouse gas. By using blended cement, 50% content of Portland cement can be replaced thereby diminishing carbon dioxide emissions and the amount of energy required to produce cement. In terms of financial, material used in making the blended cement such as fly ash are available abundantly almost at free of cost. For petroleum industry, this new blended cement is expected to help oil and gas company to minimize risk of such as low compressive strength by introducing more stable and strong cement, low permeability and low thickening time.

1.3 Objectives and Scope of the project

1.3.1 *Objectives*

Some objectives set and satisfy the scopes of study that have been highlighted, which are relevant to the requirement to complete the FYP. The objective of this project is to study properties of new blended cement such as compressive strength, thickening time and fluid loss.

1.3.2 *Scope of Project*

The scope of study is mainly creating 5 sets of blended cement model and record the data after doing compressive strength test, thickening time test, fluid loss test and microstructure testing. For the first part of the project, research is been done using journal papers and articles. After identifying the materials that suitable for making the new blended cement, the compositions of the material must be decided. The compositions are starting with 50% for cement class G, 0% for silica fume and 50% for fly ash class F. The compositions are increasing by 12.5% increment for each set. This blended cement will be compared to the conventional cement in terms of compressive strength, thickening time and fluid loss. Composition for conventional cement is 100% cement class G. Standard used for blending cement is using API specification 10A and ASTM C-618 for fly ash. All the experiments will be conducted in the cement lab supervised by lab technicians.

1.4 Relevancy of Project

The approach of this research would involve experimental studies to achieve mentioned objectives. Using knowledge from Drilling and Production subjects and also guidance from supervisor and lab technician, this project would be possible to succeed. This project also could be one of a stepping stone to work in oil and Gas Company in future planning by introducing new blended cement to be implemented in Malaysia wells.

1.5 Feasibility of Project

This project will be conducted based on experimental in the lab and also require some basic software such as Microsoft Excel ® 2011. These materials that will be use during this project are also available abundantly in Malaysia. Furthermore, these materials also will not affect health and safety if conducted safely. By referring to the Gantt chart, this project is possible to finish in time since every tools and equipment already available in cement lab.

Chapter 2
LITERATURE REVIEW

2.1 Theory

Blended Cement is obtained by mixing OPC with mineral admixtures or additives like fly-ash, slag or silica fumes [4]. Blended cements are not being considered superior as compared to conventional OPC category of cements. The advantages of using the blended cement can be divided in two categories which are technical advantages and environmental advantages.

For technical advantages, it reduces water demand and therefore water content ratio can be reduces. It also improves workability for the same water content. The blended cements are finer as compared to OPC therefore the permeability of concrete is less. As permeability is reducing, this proved to increase durability.

In case of environmental, blended cement prove to be energy saving. As stated above, blended cements are obtained by adding admixtures with OPC. The energy, which would have been used for production of OPC, is thus saved. The used of mineral admixtures can save energy and lower the emissions from cement plants thus conserving the precious minerals like lime stone, clay and so on [5]. By reducing the production of the cement, pollution is also controlled as cement is an extensive product. Based on article in website 7% of total present pollution is only due to cement production which can proportionately be reduced if more blended cement id used [6].

The materials proposed to enhance cement thus making new blended cement oil-well are silica fume, fly ash, fluid loss additives, retarded and cement class G. The test will be conducted by four main parts including the fluid loss test, the thickening time test and compressive strength tested at simulated reservoir condition.

2.2 Materials

2.2.1 *Oil Well Cement*

Oil well cements are manufactured in accordance with the American Petroleum Institute Specification API Standard 10A. Cements are designated by eight classes. The classification for each is based on the pressure-temperature thickening time encountered at specified depths in the primary cementing of casing in wells. Cement slurries may often be exposed to bottom-hole pressures in excess of 140 MPa (20, 000 psi) and temperatures approaching 120^{o}C (250 F) [7]. Oil well cements are made from the same basic ingredients as regular cements; however, a certain properties are altered so that the cements can performs as intended at the higher temperatures and pressures encountered in deep wells. Admixtures and other ingredients such as sand, bentonite, pozzolan, and diatomaceous earth, are incorporated into the mixture for the purpose of controlling its fluid properties; organic compounds are added to control its setting time. API class cement is shown in Table 2.1 below:

Table 2.1: API classes cement [7].

API Class	Operating Temperature (F)	Suitability
A	80-170	Good for 0-6000ft depth. Used when special properties are not required
B	80-170	Good for 0-6000ft depth. Used for moderate to high sulphate resistance
C	80-170	Good for 0-6000ft depth. Used for moderate to high sulphate resistance and when high early strength is required.
D	170-230	Good for 6,000-10,000ft depth. Used for moderate to high sulphate resistance and moderately high temperatures and pressures.
E	170-290	Good for 10,000-14,000ft depth. Used for moderate to high sulphate resistance and high temperatures and pressures.
F	230-320	Good for 10,000-16,000ft depth. Used for moderate to high sulphate resistance and extremely high temperatures and pressures.
G	80-200	Good for 0-8,000ft depth. Used for moderate to high sulphate resistance. Has improved slurry acceleration and retardation
H	80-200	Same as class G

2.2.2 *Silica Fume*

Silica fume is a by-product of the silicon and ferrosilicon metal manufacturing process. This finely divided, glassy powder results from the condensation of silicon oxide gas. Particles are 100 times smaller than the typical particles of Portland cement. **Silica fume** is usually categorized as supplementary cementitious material. This term refers to materials that are used in concrete in addition to Portland cement

[8]. These materials can exhibit the properties of Pozzolanic, cementitious and combination of both. Pozzolanic is when the materials will not gain strength when mixed with water. For cementitious, it will gain strength when mixed with water. Silica fume is often known to by other names such as condensed silica fumes, micro silica and volatilized silica. Furthermore, there are several materials that are physically and chemically quite similar to silica fume for instance, precipitated silica, fumed silica, gel silica, colloidal silica, silica flour and silica dust [9]. The primary chemical and physical properties of silica fume are Table 2.2 and 2.3:

Table 2.2: Chemical properties [10].

Chemical Properties of Silica Fume
Amorphous
Silicon dioxide > 85%
Trace elements depending upon type of fume

Amorphous is a term called when the silica fume is not a crystalline material. A crystalline material will not dissolve in concrete, wish must occur before the material can react. There are materials that chemically similar to silica fume which is sand. For silicon dioxide, this is the reactive material in silica fume. Lastly, for trace elements, there may be additional materials in the silica fume based upon the metal being produced in the smelter from which the fume was recovered. Basically, these materials have no impact on the performance of silica fume in concrete [10].

Table 2.3: Physical properties [10].

Physical Properties of Silica Fume	
Particle size (typical)	<1µm
Bulk density : (as-produced): (densified):	 130 to 430 kg/m^3 480 to 720 kg/m^3
Specific gravity	2.2
Specific surface	15,000 to 30,000 m^3/kg

Adding silica fume brings large amount of very small particles to a concrete/cement mixture which fills in the spaces between cement grains. This phenomenon is frequently referred to as particle packing or micro-filling [11]. Even if silica fume did not react chemically the micro-filler effect would bring significant in the nature of the concrete. Below is the table that shows the comparison between silica fume and other materials.

Table 2.4: Comparison of silica fume and other materials [10]

Comparison of Size of Silica Fume Particles and Other Materials		
Material	**Nominal Size**	**SI units**
Silica fume particle	N/A	0.5 µm
Cement grain	No. 325 sieve	45 µm
Sand grain	No.8 sieve	2.36 mm
Coarse aggregate particle	¾ inch sieve	19.0 mm

Based on Table 2.4, silica fume have smaller dimension compared to the other material which make it a very suitable materials to fill in the void particles. Because of its very high amorphous silicon dioxide content, silica fumes are a very reactive Pozzolanic material in concrete. As the Portland cement in concrete begins to react chemically, it releases calcium hydroxide. The silica fumes reacts with this silica hydroxide to form additional binder material called calcium silicate hydrate, which is very similar to the calcium silicate hydrate formed from the Portland cement [10]. It is largely this additional binder that gives silica-fume cement its improved hardened properties. In case for cementing in reservoir, permeability also should be concern about since high permeability can damage the cement since the durability of cement id directly related to its permeability. By lowering w/cm and adding silica fumes, the permeability of the concrete can be reducing.

2.2.3 *Fly Ash*

ASTM C 618 outlines the physical and chemical requirements of pozzolanic materials. Pozzolanic materials include natural pozzolans (Class N) and by-product materials. Natural pozzolans are notably volcanic ashes, diatomaceous earth, calcined clay, metakolin clay and rive hull ash. By-product material is most commonly regards as fly ash, classified as either Class F or Class C reflecting a difference in chemical composition and origin. Class F fly ashes possess largely pozzolanic properties. Class C fly ashes generally possess cementitious as well as pozzolanic properties. **Fly ash** has been used in roadways and interstate highways since the early 1950s. Furthermore, in January 1983, the Environmental Protection Agency published federal comprehensive procurement guidelines for cement and concrete containing fly ash to encourage the utilization of fly ash and establish compliance deadlines [12]. Work at the University of California published in 1937 served as the foundation for specifications, methods of testing, and use of fly ash for this application. This work concluded "That where available, fly ash's fineness and composition is suitable and can be used with technical benefits and economy to replace 20 to 50% of the amount of Portland cement that otherwise would be required to produce concrete of specific strength and durability."[12] Fly ash is the finely divided residue that results from the combustion chamber by exhaust gases and is produced by coal-fired electric and steam generating plants. Fly ash particles are generally spherical in shape and range in size from 10 µm to 100 µm and typically consist of most Silicon Dioxide (SiO_2). There are two forms which are amorphous and crystalline. Amorphous is rounded and smooth and crystalline is sharp and hazardous. The main constituent of fly ash is silica [12]. Fly ash is finer than Portland cement in term of size particles. Fly ash consists of silt-sized particles which are generally spherical and ranging in size between 10 – 100 µm. These small glass spheres improve the fluidity and workability of cement. Fineness is one of the important properties contributing to the Pozzolanic reactivity of fly ash [12].Based on ASTM C-618, there are two classes of fly ash which are Class F fly ash and Class C fly ash [13]. The difference between those two is in terms of amount of calcium, silica, alumina, and iron content in the ash. Below is the sample oxide analysis on class F fly ash and class C fly ash:

Table 2.5: Sample oxide analysis [14].

Compounds	Fly Ash Class F	Fly Ash Class C	Portland Cement
Silicon Dioxide (SiO_2)	55%	40%	23%
Aluminum Oxide (Al_2O_3)	26%	17%	4%
Iron Oxide (Fe_2O_3)	7%	6%	2%
Calcium Oxide (CaO)	9%	24%	64%
Magnesium Oxide (MgO)	2%	5%	2%
Sulfur Trioxide (SO_3)	1%	3%	2%

Based on Table 2.5, the content of calcium oxide for fly ash class C is higher than fly ash class F. So fly ash class C has cementitious properties and will not dependable to other binder material unlike fly ash class F.

Class F fly ash is produced by burning of harder, older anthracite and bituminous coal and consist primarily of an alumino-silicate glass with quartz, ullite and magnetite also present. This class also contains less than 20% CaO. Class F fly ash require a cementing agent, such as Portland cement with the presence of water in order to react and produce cementitious compounds [13]. Class F fly ash can be used when blended with lime, Portland cement or cement kiln dust. Typical proportions for the class F fly ash lime blends are 2-8% blended with 10-15% class F fly ash. Also, 0.5 -1.5% Portland cement can be blended with Class F fly ash to produce the stabilizing agent. The stabilization of aggregate bases provides some advantages such as add significant strength and durability and reduces project cost since fly ash is a relatively cheaper material.

Class C fly ash is produced from the burning of younger lignite or sub bituminous coal, in addition to having Pozzolanic properties and self-cementing properties which means Class C fly ash does not require an activator unlike Class F. Furthermore, Class C fly ash contains more than 20% CaO. Advantages for self-cementing fly ash generally include stabilization of the soil to improve the engineering properties such as increase in strength or sub grade capacity, drying of the soil to facilitate compaction and treatment of the soil to reduce shrink-swell potential [15].

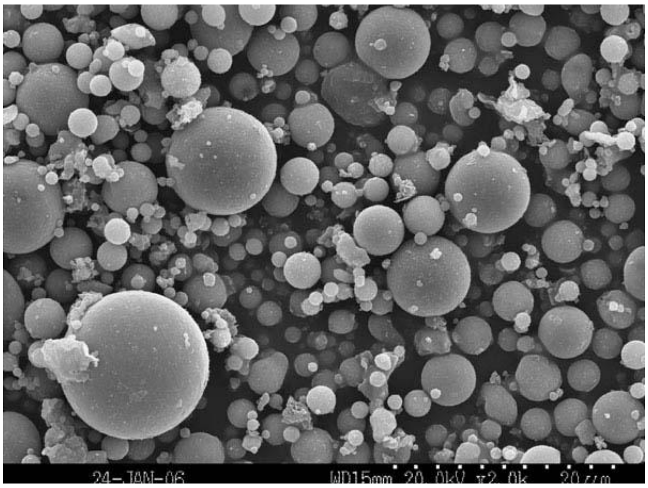

Figure 2.1: Fly ash particles viewed at 1000x magnification [14].

2.3 Additives

2.3.1 *Fluid-loss Control Additives*

As the slurry passes permeable strata, fluid is filtered out of the slurry by differential pressure, depositing a layer of solids (filter cake) on the formation face. The loss of this filtrate alters the physical properties of the slurry. Cement slurry with inadequate fluid loss control can possess inconsistent slurry properties. Thickening time is shortened, viscosity is increased, and mud displacement efficiency is diminished. Inadequate fluid-loss control can also promote gas migration, formation damage, and be responsible for unsuccessful remedial operations [16].

In order to avoid poor zonal isolation or poor cement bonding, fluid loss agents are incorporated to help the cement slurry maintain its fluid and control the rate of fluid loss to permeable strata, thus insuring an adequate water-to-cement ratio. Fluid loss agents function primarily by promoting the deposition of a low permeable filter cake. The fluid-loss control additives provide job design simplicity, better control of pumping operation, and more consistent slurry properties.

Fluid loss control is also critical when the slurry is being pumped in a narrow annular space. Uncontrolled water loss contributes to the deposition of a thick, permeable filter cake which results in bridging process that leads to complete annular blockage.

Filter cake development can be viewed as a process of filtration. The cement particles suspended in the slurry, under differential pressure, are filtered by permeable strata. The deposited solids form a filter cake whose structure is influenced by particle size, particle charge, packing efficiency of the particles and degree of particle compression. Once this framework of solids is built, further reduction in filter cake's permeability is dependent on the action of the fluid loss polymers. The polymeric effects responsible for the development of a low permeability filter cake from the framework of cement solids include [16]:

i. Polymeric attachment to a cement surface and extension of the polymer into the pore space through which the filtration must pass. The water binding property of the polymer then allows them to efficiently lug the interstitial spaces of the filter cake
ii. Polymers located within the pore network mechanically plug the pore spaces resulting the fluid loss control
iii. The viscosification of the interstitial fluid by the polymeric material.

Below is the table that shows three (3) ways in which water reducers can be apply:

Table 2.6: How fluid loss can be used [17].

How fluid loss can be used	
Applications	**Benefits**
Add water reducer without changing mix proportions	Higher slump with no change in water-cement ratio or cement content
Add water reducer, decrease water content and increase aggregate content to keep clump, cement content and yield (mix volume) constant	Lower water-cement ratio (higher strength) with no change in slump or cement content
Add water reducer, decrease water and cement content and increase aggregate content to keep slump water-cement ratio and yield constant.	Lower cement content with no change in slump or water-cement ratio.

Based on Table 2.6, by choosing the third method, we can lower the cement content with no change in water-cement ration. This can results in low water requirement for mixing with the cement which can be beneficial for fluid loss test.

2.3.2 *Retarders*

Retarders are added to cement slurries to lengthen the thickening time (TT) and/or to slow down the hardening process. They are often used to lengthen the thickening time at moderate to high temperatures, to offset the shortened setting time caused by certain other chemicals, such as extenders [16].

The most commonly used retarders in cement slurries are sodium and calcium salts of lignosulfonic acids. Lignosulfonates are polymers derived from wood pulp; therefore, they are usually unrefined and contain various amounts of saccharide compounds. Purified lignosulfonates lose much of their retarding power, the retarding action of these additives is often attributed to the presence of low molecular- weight carbohydrates. Lignosulfonate retarders are effective to about 250 F (122 C) bottom-hole circulating temperature (BHCT) [16].

Saccharide compounds (so-called sugars) are good retarders of cement slurries. They are not commonly used in well cementing because the degree of retardation is very sensitive to small variation in concentration.

Many inorganic compounds retard the hydration of Portland cement. These compounds are [17]:

1. Acids and salts of: boric, phosphonic, hydrofluoric and chromic (borax is commonly used as “retarder aid”. It has the ability to extend the effective temperature range of most lignosulfonate retarders to as high as 600 F (315C), however, it can be detrimental to the effectiveness of cellulosic and polyamine fluid-loss control additives).
2. Oxidizers: zinc and lead (zinc oxide is sometimes used for retarding thixotropic slurries, because it does not affect the slurry rheology, nor does it affect the hydration of C3Agypsum system).
3. sodium chloride (table salt): concentration > 20% BWOW

Most retarders will cause the viscosity of the cement slurries to increase.

2.4 API Mixwater Requirements for API Cement

Mixwater is the water which is used to make up the cement slurry. Too much mixwater can result in failure of the cement to set into a strong, impermeable cement barrier. Too little mixwater results in increase in slurry density and viscosity, decrease of pumpability and lesser volume of slurry produced from each sack of cement.

Table 2.7: API mixwater requirements [9].

Class	Mixwater (gal/sack)	Slurry Weight (lbs/gal)
A	5.2	15.6
B	5.2	15.6
C	6.3	14.8
D	4.3	16.4
E	4.3	16.4
F	4.3	16.2
G	5.0	15.8
H	4.3	16.4

2.5 Cementing Calculations

Cementing calculations are an essential part of the designing stage of a cement job. Slurry calculations used in conjuction with fill-up calculations, the slurry weight, slurry volume, and water requirement calculations determine the proper amount of dry-blended cement and water needed for a particular job. Calculation aids are applied when all additive concentrations except salt are based on the weight of cement. When using blended cement systems, the additives are based on the weight of the mixture of cements. Additives used in low concentrations do not appreciably affect calculations and can generally be ignored while additives used in larger concentrations are included in the calculations such as barite, silica sand Thixad and salt [18].

Weight of materials (lb/sk):

$$94 + (8.33 \times vol\ of\ water, gal) - (\%\ additive \times 94)$$

Volume of slurry (gal/sk):

$$\frac{\frac{94lb}{sk}}{SG_C \times \frac{8.33lb}{gal}} + \frac{W_A}{SG_A \times \frac{8.33lb}{gal}} + V_w$$

If the desired density is known, the water requirement can be obtained using material balance equation

$$\rho_1 V_1 = \rho_2 V_2$$

Number of sacks of cements:

$$N_C = \frac{V_S\,(ft^3)}{Y_s\left(\frac{ft^3}{sk}\right)}$$

Total weight of additive required (lb)

$$W_A = W_A \times N_C$$

Total volume of water required (gal)

$$V_W = V_W \times N_C$$

Amount of high density additive required per sack of cement to achieve a required cement slurry density

$$x = \frac{\frac{\rho_s \times 11.207983}{SG_C} + (\rho_s \times CW) - 94 - (8.33 \times CW)}{\left(1 + \frac{AW}{100}\right) - \left(\frac{\rho_s}{SG_A \times 8.33\,\frac{lb}{gal}}\right) - \left(\rho_s \times \frac{AW}{100}\right)}$$

x=additive required, lb per sack of cement

p_s*=required slurry density, lb/gal*

CW=*water requirement of cement*

AW=*water requirement of additive*

SG_c=*specific gravity of cement*

SG_A=*specific gravity of additive*

Chapter 3
METHODOLOGY

3.1 Research Methodology

The project is mainly divided into two (2) different parts, which are FYP II and I. The tasks proposed for both FYP II and I are shown in Table 3.1. A thorough study has to be conducted in order to make this research successful. In order to achieve the objectives of the project, researches have been done on numerous resources including articles, journals and internet. The details of progress to finish this project can be referred to the figure 3.12. The important activities need to be done during the FYP period are:

Table 3.1 : FYP planning.

No.	Task / Activity	Objectives of the task(s)	Remarks
1.	Preliminary research	Obtaining basic understanding for extended proposal preparation	Conducted from Week 1 till Week 5 on semester May 2011.
2.	Preliminary work	To provide basic requirement for the project. • Lab scheduling. • Obtaining permission for equipment usage. • Understanding the procedure of the experiment. • Preparing the extended proposal.	By the time of this proposal submitted, only approval from laboratory executive is still in process.
3.	Repetition of previous work done.	To understand the system mechanism. To observe the effect of human error in conducting the experiment. To find suitable improvement for the system.	Expected to commence after Mid-Semester break.
4.	Data analysis	To observe the outcome from the previous task. To determine the required parameter for the second experimental work.	Expected to be included in the interim report.
5.	System improvement	To provide better results compared to previous work done.	To be commenced during FYP 2.
6.	Experiment with new parameters	To conduct the experiment under new parameter compared to previous work done.	The core of the project, expected to consume most of the time.
7.	Data analysis	To analyze the data obtained from the experimental work.	To be conducted simultaneously with the experimental work.
8.	Preparation for presentation and report writing	To provide means of delivering the result from the experiment to people.	Part of project evaluation.

3.2 Flow Chart

The following flow chart explains the methodology in executing the project:

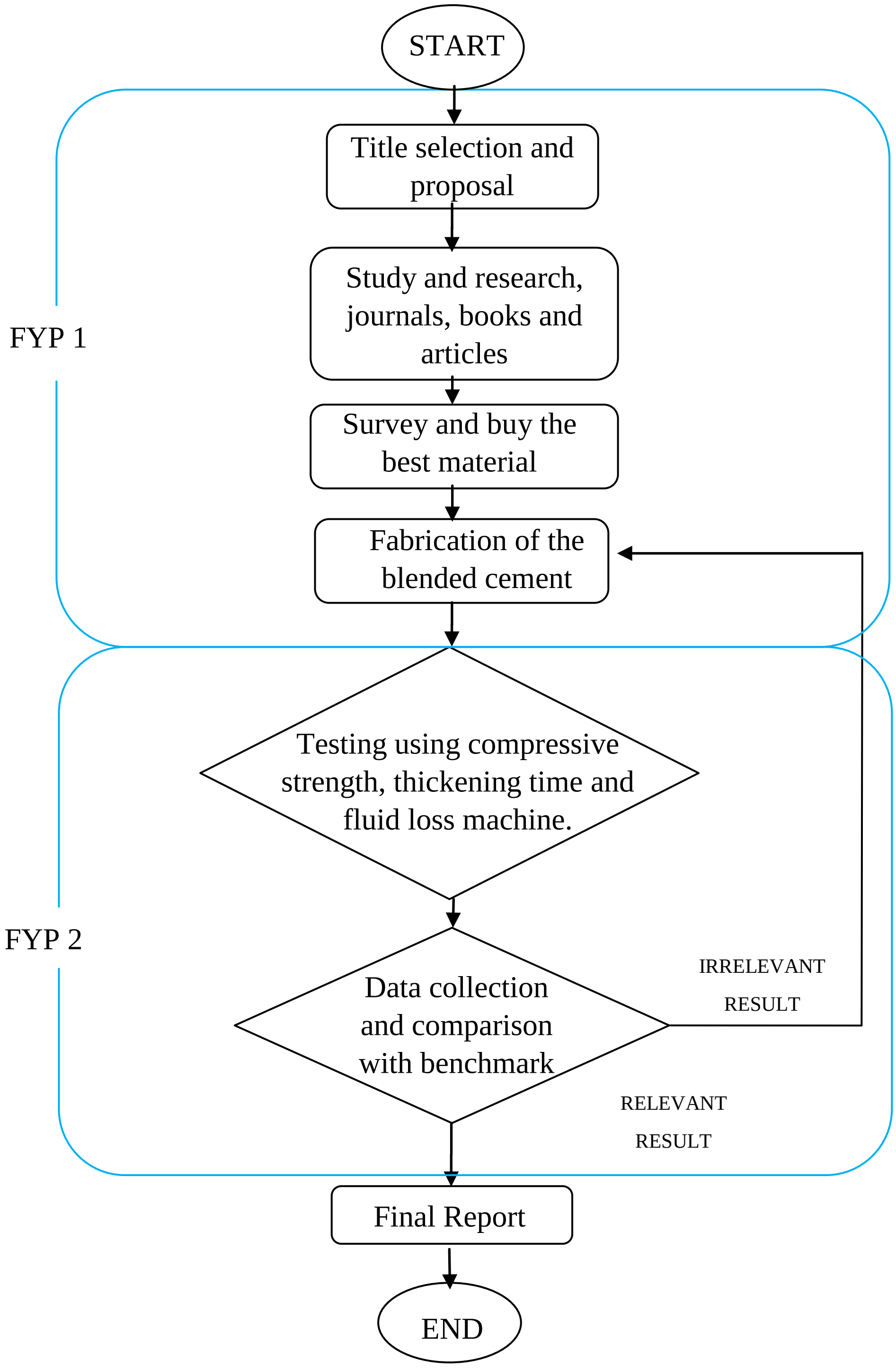

Figure 3.1 : Project methodology.

3.3 Tools and Equipment Required

3.3.1 *HPHT Curing Chamber*

HPHT Curing Chamber is utilized to prepare well cement specimens for compressive strength tests. It is necessary to determine the amount of time required for a cement to develop compressive strength so that drilling/production operations can be resumed as quickly as possible. The goal is to design slurry which can quickly develop compressive strength so that the waiting on cement time can be reduced. This HPHT curing chamber was designed to provide a means of curing cement specimens under typical down hole temperatures and pressures.

Method of operation to conduct the project using this machine is cement is poured into a special mold which produces specimens that measure 2” X 2" X 2" (5cm x 5cm x 5cm). The mold is placed into the test cell and the pressure of the test cell is increased via an air driven hydraulic pump. Test temperature is governed by a PID temperature controller which actuates the heater. After a predetermined amount of time the temperature of the test cell is reduced by the cooling system. The cement slurry shall be prepared in accordance with Section 5 of API Specification 10.Specimens are removed and the compressive strength is determined as outlined in API Specification 10.

The slurry shall be placed in the prepared molds in a layer equal to one-half of the mold depth and puddle 25 times per specimen with a puddling rod. The slurry shall be placed in all the specimen compartments before commencing the puddling operation. After puddling the layer, the remaining slurry shall be stirred by hand using a puddling rod or spatula to eliminate segregation, the molds filled to overflowing, and puddled as for the first layer. After puddling, the excess slurry shall be struck off even with the top of the mold, using a straight-edge. Specimens in molds which show evidence of leaking shall be discarded. A greased cover plate shall be placed on top of the mold. For one test determination, not less than four (4) specimens shall be employed.

General precautions, the curing chamber may be exhaust vapor results from heating the lube oil. Always wearing the safety mask will prevent inhaling the vapor. In the heating the lube oil process, the curing chamber may be also be heated. The user must wear safety gloves to prevent any burned skin.

Figure 3.2: HPHT curing chamber.

3.3.2 *Constant Speed Mixer*

The constant speed mixer are designed in accordance to API Specification 10 and have constant speed operation independent of the line voltage and rate that cement is added to the mix water. Good practice indicates that the cement be added slowly over a fifteen (15) second period as specified by the latest edition of API Specification 10. This constant speed mixer will be use to mix the silica fume, fly ash and cement class G. Below are the figures for mixer spoon and constant speed mixer.

Figure 3.3: Mixer spoon.

Figure 3.4: Constant speed mixer.

3.3.3 *Stirred Fluid Loss Tester*

Cement slurry is poured into the test cell, which is then placed into the heating jacket. The gear drive system is connected to the agitation paddle, which is dimensionally equivalent to an atmospheric consistometer paddle. The desired test temperature is maintained by a digital PID temperature controller, while the necessary pressure is applied to the cell to prevent evaporation of the liquid phase. When conditioning the cement in accordance to API Specification 10 guidelines, the paddle is rotated at 150 RPM for 20 minutes. Once the cement is conditioned, differential pressure is applied to the cell. The filtrate is collected in a back pressure receiver for 30 minutes. The API defines fluid loss as the volume (ccs) of filtrate that is collected during this 30-minute interval. Below is the picture of stirred fluid loss tester.

Figure 3.5: Stirred fluid loss tester.

3.3.4 *Thickening Time*

During cementing operations, the time required for cement slurry to set is of primary concern. Under an ideal situation, minimal time would be required to successfully pump the slurry, which immediately upon placement, begins to develop compressive strength. However, if insufficient time is allowed to fully pump the cement, it will be necessary to drill the cement remaining in the casing string. Remedial operations such as this are very costly. Conversely, cements that are successfully placed but require considerable time to cure, consume valuable rig time, which is also quite costly. Laboratory tests should be conducted under simulated reservoir conditions to examine the actual thickening time of the slurry. The HPHT Consistometer was specifically engineered to determine the thickening time of well cements under simulated downhole pressures and temperatures.

Method of operation is cement is mixed and poured into the slurry cup assembly. The slurry cup is placed into the test vessel and pressure is increased via an air-driven hydraulic pump. The API defines 100 bc as 2,080 gm-cm of torque. Consistency is measured by the amount of torque the slurry exerts on an API-approved paddle. A PID temperature controller governs an internal heater, which maintains the necessary temperature profile, while a magnetic drive mechanism rotates the slurry cup assembly at 150 RPM. A potentiometer controls an output voltage, which is directly proportional to the amount of torque the cement exerts upon an API-approved paddle. A multi-channel, paperless graphic recorder registers cement consistency and temperature as a function of time. Temperature and consistency are digitally displayed on screen and saved to a disk for later analysis.

3.3.5 *Compressive Strength Tester*

The properties of the cement depend on the properties of its ingredients and their proportion and it is likely to vary from mix to mix. Test must be conducted, therefore, to ensure that the cement used is in accordance with design specifications.

Figure 3.7: Compression machine.

Figure 3.6: Digital compression tester.

3.3.6 *Cement Cube Moulds*

Cube moulds are used to determine soundness of ordinary and rapid hardening Portland cement, oil-well cement, concrete and low heat Portland cement. They are mainly used for molding purposes. The moulds can only be used maximum three (3) at one time because of curing chamber limitation. Below is the picture of cube mould.

Figure 3.8: Cube moulds.

3.3.7 *Miscellaneous*

Other equipments are the things that are important during the experiment and also for safety measures. Safety in the lab is very important since most of the chemicals and equipments are dangerous. Below is the pictures for the safety tools.

Figure 3.9: Safety glove.

Figure 3.10: Mask and safety glass.

3.4 Project Activities

Table 3.2 shows that the project activities planned throughout a year which starting from week 4 until week 14.

Table 3.2: Project activities planned for final year project.

Activities	Starting Week	Finishing Week
Studies on possible material for blended cement-oil well and basic oil-well cement	Week 4	Week 5
Studies on possible materials that can be made into blended cement	Week 5	Week 6
Planning 4 different set of model and identifying suitable materials proportion for each set	Week 6	Week 7
Preparation for presentation and preparation for experimental/testing	Week 8	Week 9
Laboratory testing	Week 9	Week 10
Laboratory testing and preparation for final report	Week 10	Week 12
Report documentation	Week 12	Week 14

3.5 FYP 2 Gantt Chart

No	Detail/Work	1	2	3	4	5	6	7		8	9	10	11	12	13	14
1	Continue studies on possible material for blended cement-oil well and basic oil-well cement															
2	Studies on possible materials that can be mix into blended cement															
3	Planning two (20) different set of model and identifying suitable materials proportion for each set								Mid-Semester Break							
4	Preparation for presentation and preparation for experimental/testing															
5	Laboratory testing for benchmark samples															
6	Laboratory testing for blended cement and preparation for progress report and poster															
7	Report documentation															

Figure 3.11: FYP 2 gantt chart.

3.6 FYP 2 Key Milestone

Table 3.3: FYP 2 key milestone.

No	Detail/Work	1/9	5/9	12/9	19/9	26/9	3/10	10/10	Mid-Semester Break	17/10	1/11	8/11	21/11	1/12	5/12	12/12
1	Ordering class G cement from Lafarge Johore															
2	Checking where silica fume and fly ash can be taken															
3	Discussion with supervisor regarding suitable composition for blended cement															
4	Silica fume and fly ash taken from Civil block															
5	Experiment started for sample benchmark and blended cement for compressive strength															
6	Experiment started for sample benchmark and blended cement for compressive strength, thickening time and fluid loss															
7	Report documentation															

Chapter 4
RESULTS & DISCUSSIONS

4.1 Data Gathering and Analysis

The expected result that is targeted to be achieved at the end of this project is to complete the benchmark samples and blended cement samples. The parameters that affect of the performance and consistency of the blended cement also will be investigated in order to get consistent results. Several experiments need to be done in order to find the best composition of silica fume, fly ash and class G cement that affect the compressive strength, thickening time and fluid loss. The samples benchmark will be based on the conventional cement usually used in Malaysia basin.

4.1.1 *Cement Samples Benchmark Test*

This experiment will test the samples benchmark with additives (fluid loss additives, retarded). After curing for 24 hours, the samples will be cure in the water for four (4) days. These four samples will be tested for compressive strength. The result of this experiment is tabulated in Table 4.1. At this level, the longer the samples cured in the curing chamber, the higher strength will be produced. In order to standardized, all samples will undergo curing period for 24 hours.

Table 4.1: Compressive strength test for sample benchmark.

Sample Benchmark with additives		
No	**Result**	
1	45.02 MPa	6531.5 psi
2	42.73 MPa	6199.3 psi
Average	**43.9 MPa**	**6365.4 psi**

Figure 4.1: Benchmark samples after
compressive strength test.

4.1.2 *Blended Cement Composition*

After completing the fabrication and testing for the samples benchmark, the composition for the blended cement is done. Four (4) compositions are prepared in order to test which composition is the best. The composition is tabulated in the Table 4.3.

Table 4.2: Composition for blended cement (%).

Sample	Class G Cement	Silica Fume	Fly Ash Class F
1	50%	0%	50%
2	50%	12.5%	37.5%
3	50%	25%	25%
4	50%	37.5%	12.5%
5	50%	50%	0%

Table 4.3: Composition for blended cement (gram).

Sample	Class G Cement	Silica Fume	Fly Ash Class F
1	191.4 g	0g	191.387g
2	191.4g	47.9g	143.55g
3	191.4g	95.7g	95.7g
4	191.4g	143.6g	47.9g
5	191.4g	191.4g	0g

4.2 Compressive Strength Test

Compressive strength cured at same pressure and temperature according to the Section 5 API Specification 10A. The results for the compressive strength test are shown in Table 4.4.

Table 4.4: Compressive strength of blended cement (cement/silica fume/fly ash).

	100/0/0 psi	**50/0/50 (psi)**	**50/12.5/37.5 (psi)**	**50/25/25 (psi)**	**50/37.5/12.5 (psi)**	**50/50/0 (psi)**
1st result	6531.5	5208.4	5814.8	5721.9	7473.1	6284.8
2nd result	6199.3	5385.37	5663.9	5681.3	8130.3	5791.6
Average	6365.4	5296.9	5739.4	5701.6	7801.7	6038.2
Standard deviation	234.9	125.1	106.7	28.7	464.7	348.8

Figure 4.2: Sample 1 (50/0/50).

Figure 4.3: Sample 2 (50/12.5/37.5).

Figure 4.4: Sample 3 (50/25/25).

Figure 4.5: Sample 4 (50/537.5/12.5).

Figure 4.6: Sample 5 (50/50/0).

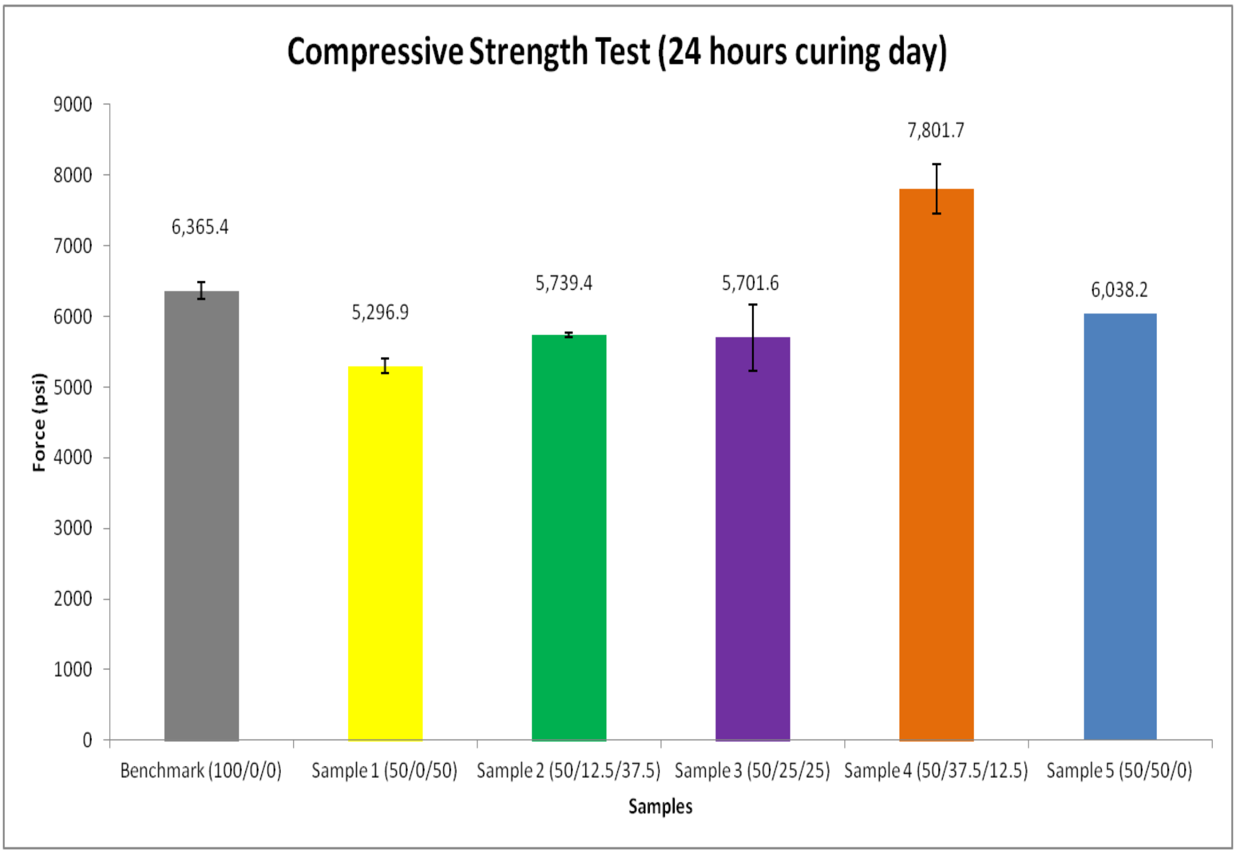

Figure 4.7: Compressive strength for blended cement (cement/silica fume/fly ash).

From Table 4.4, it is observed that the sample 2 still not exceeding the benchmark's compressive strength value. However, sample 3 and sample 4 are expected to exceed the benchmark's compressive strength value because of high silica in that particular composition. The results of the new blended cement are compared to the previous model's results and it is tabulated as Table 4.5 below:

Table 4.5: Comparison between benchmark and sample 4 blended cement (50/37.5/12.5).

Sample	Compressive Strength
Benchmark (100/0/0)	6302.2 psi
Sample 4 (50/37.5/12.5)	7801.7 psi

From the results, it is clear that blended cement with more silica fume content have more strength compared to the conventional cement. With high pressure and temperature, high content of tricalcium aluminate, a very fast rate of reaction during hydration and a very fast setting time explains why it has higher strength compared to others.

4.3 Thickening Time Test

Thickening time tests are conducted according to the API Specification 10A. The results for the thickening time are tabulated in the Table 4.5 below:

Table 4.6: Thickening time for blended cement (cement/silica fume/fly ash).

Sample	At 40 Bc (min)	At 70 Bc (min)	At 100 Bc (min)
Sample 1 (50/0/50)	63	89	92
Sample 2 (50/12.5/37)	73	103	130
Sample 3 (50/25/25)	89	119	149
Sample 4 (50/37.5/12.5)	112	142	169
Sample 5 (50/50/0)	99	129	159
Benchmark (100/0/0)	88	101	114

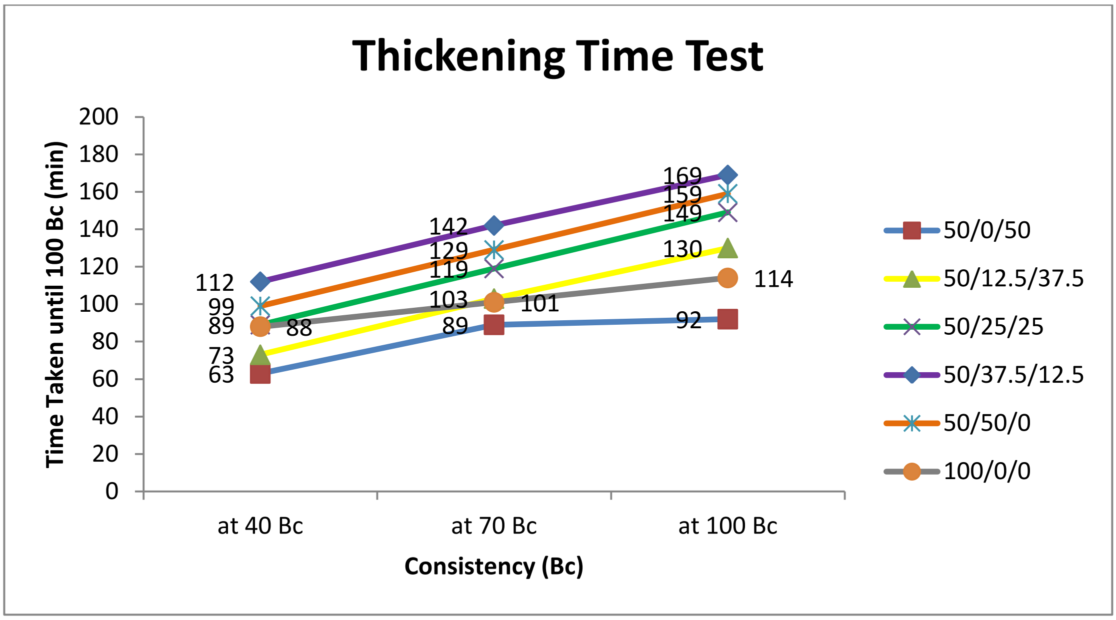

Figure 4.8: Thickening time for blended cement (cement/silica fume/fly ash).

Table 4.6 shows the results of the thickening time tested 8000 feet and 52 degree Celsius. It was found that blended cement will have higher thickening time than conventional cement. With the difference in the content of fast reacting substance, that is tricalcium aluminate explains why each cement will set at different time. Chemically, combination of silica fume and fly ash contains less tricalcium aluminate than conventional cement thus resulting in higher thickening time.

The amount of tricalcium present in the cement composition has an influence on the setting time of the cement and the amount of fluid loss added also helped to delay the thickening time in the blended cement by delaying the contact of cement grains with water to undergo the hydration process. The longest thickening time is by sample 4 as tabulated in the Table 4.7 below:

Table 4.7: Comparison between benchmark and sample 4 blended cement (50/37.5/12.5).

Sample	**Thickening Time (min) at 100 Bc**
Benchmark (100/0/0)	114
Sample 4 (50/37.5/12.5)	169

4.4 Fluid Loss Test

Fluid loss tests are conducted according to the API Specification 10A. The results for the thickening time are tabulated in the Table 4.8 below:

Table 4.8: Fluid loss results for blended cement (cement/silica fume/fly ash).

Sample						
Time	**Fluid Loss (ml)**					
	100/0/0	**50/0/50**	**50/12.5/37.5**	**50/25/25**	**50/37.5/12.5**	**50/50/0**
0.25	94	28	20	15	5	11
0.5	97	40	33	21	8.5	23
1	100	60	54	33	13.5	26.2
2	112	89	67	45	21	36
5	117	125	99	67	37.5	45
10	119	137	112	77	59.5	71
15	200	141	124	89	79	82
20	204	143	131	91	88	91
25	208	144	139	97	91	99
30	210	145	141	100	94	112

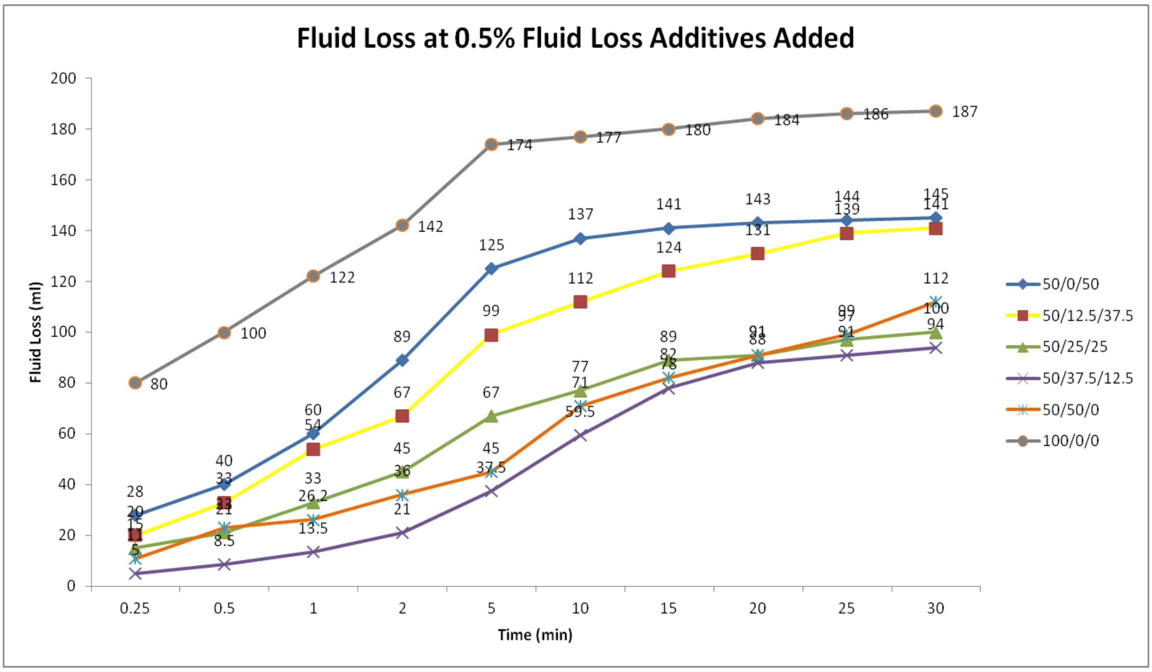

Figure 4.9: Fluid loss for blended cement (cement/silica fume/fly ash).

From the results in Table 4.8, in term of fluid loss, new blended cement is proven to release less water as compared to the sample benchmark. It proves that, during the cement reaction and with the existing water, fine particles of fly ash and silica fume will react during the early reaction to form additional cementitious material of tricalcium silicate hydrates which filled the existing voids and thus will reduce the number of voids, and consequently reduce permeability of the cement. With higher content of tricalcium aluminate in silica fume and fly ash, it will react at a faster rate during hydration and means less water is released compared to conventional cement. So the most less water released is by sample 4 as compared with other samples and benchmark as shown in Table 4.9 below.

Table 4.9: Comparison between benchmark and sample 4 blended cement (50/37.5/12.5).

Sample	**Fluid Loss (ml)**
Benchmark (100/0/0)	210
Sample 4 (50/37.5/12.5)	94

Chapter 5
CONCLUSIONS & RECOMMENDATIONS

5.1 Conclusions

Firstly, the targeted objectives have been successfully achieved. The new blended cement (50% cement, 37.5% SF, 12.5% FA) is the highest in term of compressive strength and thickening time, and lowest in fluid loss. In terms of fluid loss, thickening time and compressive strength tested at atmospheric temperature and pressure and simulated reservoir condition, blended cement expected to have the properties suitable for the application in the oil-well cementing operations. Based on results, the increase in compressive strength of the blended cement samples tends to be higher with the increasing contents of silica fume. So, the better model is sample 4 with 18% of improvement for compressive strength, 32% improvement for thickening time and 55.2% improvement for fluid loss.

In the bore hole, the cement is subjected to complex triaxial loading and the failure stresses maybe considerably different from those observed in the standard compressive strength tests. Moreover, compressive strength measurement provides no guide to the shear strength of the casing/cement or the casing/formation bond [19].Though compressive strength provides no lead for the shear strength of the bonds, it gives some hint of the strength to hold formation pressure and it is better if adequate strength develops in less than 24 hours of cementing of oil-wells.

5.2 Recommendations

From the result obtained, I would like to recommend a research on deep water or high pressure high temperature can be conducted since this project only cover for shallow water and primary wells. Thus, blended cement capability can be expanded and might be used for other type of wells. Besides that, I would to recommend a site visit to plant or material lab in order to get better view of common blended cement used nowadays. For that, useful tips or advice can be collected from engineers and lab technician there hence, can be used for this project.

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