

**CEMENTING DESIGN FOR DEEP CARBON DIOXIDE (CO₂)
INJECTION WELLS**

by

NURUL IFFAH BINTI MUHAMMAD GARIB

DISSERTATION

Submitted to the Geoscience & Petroleum Engineering Programme
in partial fulfillment of the requirement
for the Degree
Bachelor of Engineering (Hons)
(Petroleum Engineering)

Universiti Teknologi PETRONAS
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Perak Darul Ridzwan

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CERTIFICATION OF APPROVAL

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May 2011

CERTIFICATION OF ORIGINALITY

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

A handwritten signature in black ink, appearing to read 'Nurul Iffah Muhammad Garib', is written over a horizontal line.

NURUL IFFAH MUHAMMAD GARIB

ABSTRACT

Deep carbon dioxide injection is one of the latest studies in enhancing the oil recovery for deep well around the globe. However, Carbon Dioxide (CO₂) has the tendency to corrode and react with the Portland cement downhole. This eventually will cause severe leaking behind casing, reduce the injectivity and shorten the life of the well. Meanwhile in deeper well, the high pressure and temperature will offer problems in fluid loss rate, thickening time and unstable cement development which will shrink the potential of the cement to support the casing and seal fluids from flowing behind the casing. Due to the relatively higher pressure (<10000 psi) and temperature (250°F to 350°F), an adequate thickening time, sufficient fluid loss, faster development of compressive strength and better CO₂ resistance are expecting from the system in order to suit the condition. This paper is mainly on determining the composition of silica fume, fillite and superplasticizer that fit the provision as well as evaluating the physical and chemical properties of the new system (thickening time, fluid loss, CO₂ corrosion rate and compressive strength development). Introducing 3 new additives into the system explicitly silica fume, fillite and superplasticizer is believe to give a better performance than the neat cement. Silica fume is a common additive for deep well system and trusted to improve the strength of the sheath during the CO₂ attack. Meanwhile it is to say that fillite has the potential to replace the water required in the system and superplasticizer is added to help in reducing the water content of the cement. Cement class G is still used instead to proof that the cement is feasible for the acidic environment which can last for more than 30 years downhole alongside in improving the injectivity performance of a well. After a series of intense laboratory work, the new slurry system is proven to be better in reducing the water content which contributed to higher resistance during CO₂ attack, superior fluid loss control and strength development as well as longer thickening time that will allow deeper and longer pumping distance.

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NOMECLATURE

C_{cd} = casing displacement capacity

C_{csg} = casing capacity

C_h = hole capacity

ID_h = hole inner diameter

FL = fluid loss without blowout

FL_{ISO} = Fluid loss with blowout

L = length

OD_{csg} = casing outer diameter

PBH = bottomhole pressure

P_s = surface pressure

P_{TOC} = top of cement pressure

q = cement pumping rate

R_{pu} = pressure rate-up

R_{pd} = pressure rate-down

$R_{\Delta T}$ = temperature gradient

$R_{(\Delta T)_a}$ = temperature gradient in annulus

t = blowout time

t_d = displacement time

t_a = annulus displacement time

T_{PBHC} = bottomhole temperature

T_{SS} = surface temperature

T_{TOC} = top of cement temperature

V_a = annular volume

V_{ann} = annular volume

V_p = pipe volume

V_{st} = volume of shoe track

V_f = collected filtrate at blowout time

V_f = total volume

CHAPTER 1

INTRODUCTION

1.1 Background of Study

This study is to nurture the research on the well design and optimizing of an injection well mainly for carbon dioxide (CO₂) Enhanced Oil Recovery, EOR methods in a deep environment. This study will enable further explorations and innovations in cementing a deep well with high pumping rate of 92 - 97% concentration of CO₂ gas [1].

Normally, a deep well will be having high bottomhole pressure (7000 psi to 10000 psi) and temperature (250°F up to 350°F) which increase the issues on the cementing properties downhole. A proper selection on the cement is crucial in avoiding severe static corrosion by the acidic gas and pumping problems in displacing the slurry to its desired setting depth. The gas converts calcium silicates in Portland cement to calcium carbonate, causing the cement to have an increased permeability and to be soluble in acid (Onan 1984) which eventually capable to caused formation damage and gas leakage over the timeline.

Portland cement Class G would be the choice in cementing the annulus between the liner/casing and the formation downhole. However, corrosion will transpire over the time and this result in further research in high-alumina cement that contains calcium aluminate as the material. A wrong choice of cement will cause a severe failure which eventually reduce the injectivity performance of the well and incurring extra cost in recomplete the well after a certain short-period of time.

1.2 Problem Statements

1.2.1 Problem Identification

Cementing job is highly dependent to the borehole conditions. At relatively higher temperature and pressure condition, the physical properties of the slurry are greatly affected especially on the thickening time and fluid loss parameters of the surface-mixed composition. Figure 1 presents the experiment result on the fluid loss rate affecting by high temperature and

pressure respectively. Clearly seen that the loss rate is directly proportional will the increment of the conditions and this can be conclude that the loss rate in deep well will be severe and need to be taken into studies.

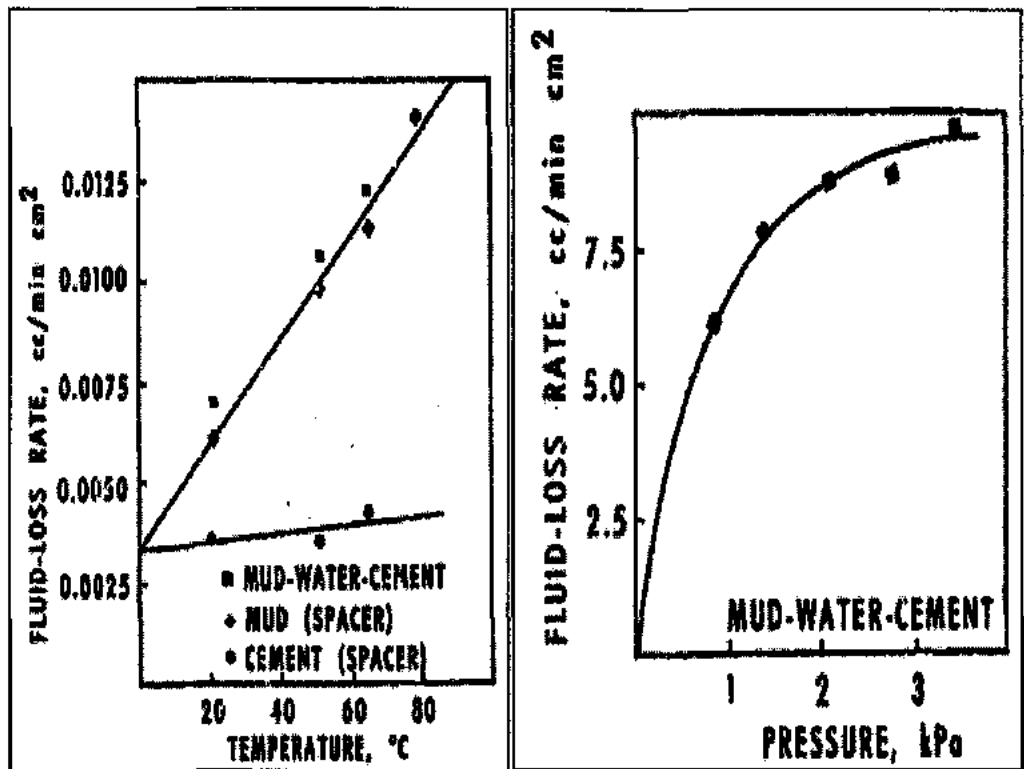


Figure 1: Experimental result of the affects of temperature and pressure on the fluid loss rate [1]

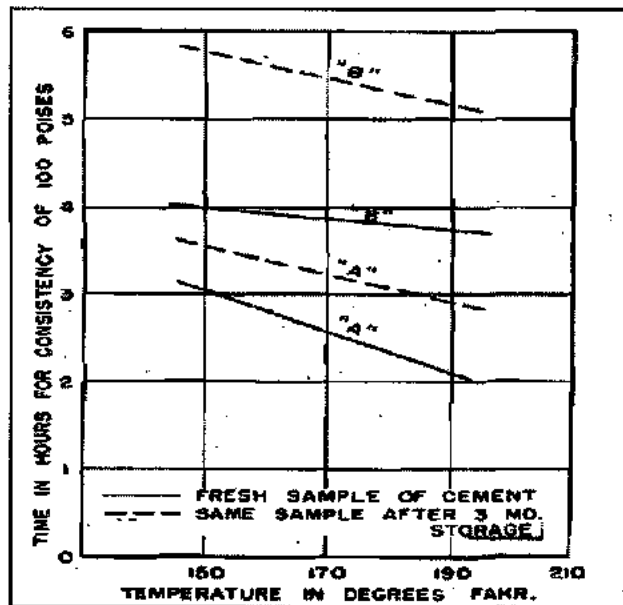


Figure 2: Effect of the temperature on to the hardening properties [1]

Furthermore, higher temperature will caused shorter period of the solidification of the system. It may be noted that by increasing the pressure will caused the slurry to be liquefied but in most cases it will shortening the time required to reach the limit of mobility. Having slurry that is not compatible with the extreme environment will cause problem in the pumping process and settling process of the cementing job.

During the hydration of the cement downhole, transition time for cement to change its phase from liquid into solid is highly dependent on the conditions of the bottomhole. During this transformation, water and gas are release out from the system and will cause holes, pores and channel settlement and causing bad and fail cementation job.

Carbon Dioxide is an acidic gas that has the ability to react with the calcium component of the Portland cement slurry compositions. Once the chemical interaction take place, the acidic substance of the gas will starts to dilute calcium to form carbonate which will cause problems such as formation damage and deflection in the cement sheath as to cause the leakage occurrence over a time of period. Figure 3 shows the graph on CO₂ concentration against the corrosion rate and the slopes prove that the higher concentration of CO₂, the faster the reaction will take place. To make it worst, since this well will be having maximum concentration of CO₂ at high temperature, the reaction time will be much shorter hence abbreviate the life of the well and its performance.

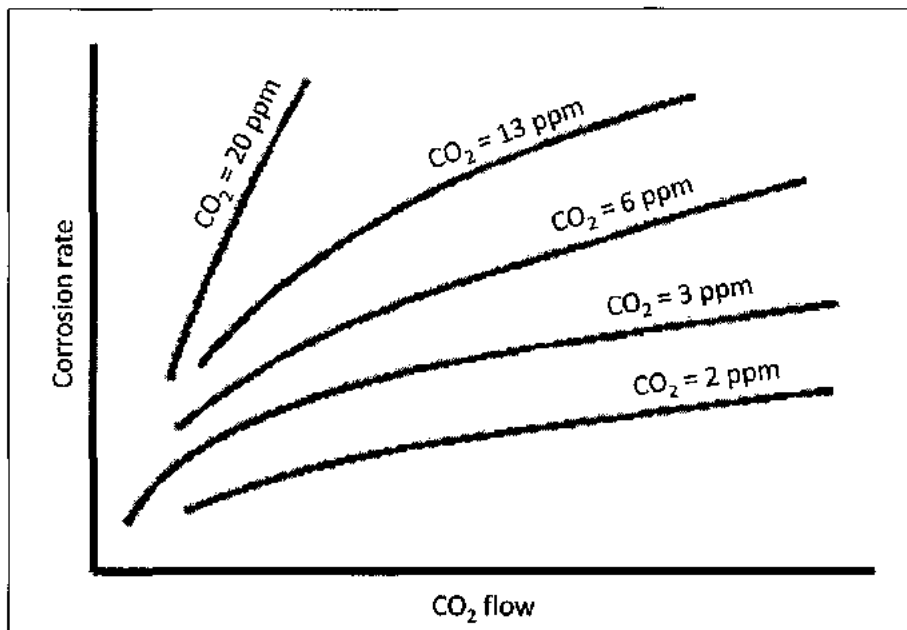


Figure 3: Impact on CO₂ concentration to the corrosion rate [11]

1.2.2 Significant of Project

Since we are dealing with a deep well and high concentration of CO₂, this project will focus more on the physical, chemical and mechanical properties of the cement slurry. In defying those problems stated, thorough experiments and studies are to be planned out in obtaining the best result and most suitable cement composition.

In providing extra thickening time for the slurry, an accurate choice of retarder and intensifier additives are to be determined comprehensively. The correct retainer and intensifier will help to provide additional minutes for the cement to be pumped to the required depth before solidify.

Moreover, an appropriate fluid loss additives selection is prompted in avoiding severe loss during the pumping of the cement into the well. Plus the cement-water ratio plays and important roles too in avoiding erosion process between water and steel substances proportional with the increase of the temperature. Reducing the ratio will eventually deduce the severity level of the fluid loss and transition time, nevertheless, will expose dilemma in pumping and mixing volumetric of the system.

High alumina cement is having a better performance in CO₂ injection environment in comparison with the Portland cement. With reason, high alumina is a processed calcium aluminate cementitious material instead of having carbonate component that will eventually reacting with the CO₂ and causing corrosion on the cementation however it is having challenges in additives selection, prevention of contamination and quality control [2]. This will defer feasibility and reliability of this project in compositing improved and more economical system for this type of well.

It is not within the scope of this study to reduce the concentration of the injection fluid. However, the right chemical composition of cement and its additives will hinder the acid gas from decaying the cement from supporting and protecting the casing for a longer lifetime. There are also several manufactured possible extra additives to be added in the cement slurry to avoid any components solute with the acid fluid.

1.3 Objectives

The objectives of this project are:

- (1) To determine the cement composition of silica fume, fillite and superplasticizer that suit for CO₂ deep injection wells.
- (2) To evaluate the performance of the cement composited.

1.4 Scope of Study

Align with the objectives of this project, the very first step of the study will be to review the physical and chemical properties of CO₂. It is necessary to understand the possible downhole problems that might contributed by the acid gas onto the cement sheath once the injection phase started. By doing this I can have a better perception on those attributes in designing proper cement slurry that can optimize the injectivity operation over a longer period of time.

In achieving the first objective, an intense reviewing and reading on the possible and tested additives is to be done. It is crucial in determining those additives because main focus would be to reduce corrosion rate but at the same time provide an adequate time for pumping, sufficient fluid loss and compressive strength in the extreme environment.

Once done, I will precede the project by doing several laboratory-works where I will be using the trial-and-error method in determine the best composition. It will start by mixing several cement composition by varying the additives percentile and determine the chemical (severity on CO₂ reaction), the physical (thickening time and fluid loss parameters) and mechanical (compression strength) properties of the slurry. The crucial part of the laboratory will be the testing on the slurry at high pressure and temperature settings.

1.5 Relevancy of Study

Having EOR as one of the most important aspects in this industry these days induced this research is relevant. With the rapid innovation and research done in increasing the produce of a particular field using CO₂ injection, this research can provoke more new technologies and innovations in upgrading the performance of the operation. Malaysia is yet to drill its first CO₂ injection well for EOR purposes therefore this will be a pilot study in designing reliable and optimum cement slurry. Since this EOR method is currently the fascinate issue to be argue and

discuss upon in most of the countries around the globe, hence this research will have the opportunity to invade the market and gain attentions for development.

Undeniable there are already CO₂ injection wells for EOR implemented in certain countries such as the United State, Canada and United Arab Emirates. However there are still plenty of rooms for improvement to optimize the performance of the well and extend the life time of those wells. Moreover, this research will be practical and cheaper in the case to renovate CO₂ injection well into other gas injections such as natural gas or nitrogen since CO₂ will have even severe reactions downhole compared to the rest gases.

Other than using for EOR purposes, this hazardous gas has been a common practice to be stored undergrounds. This is called as the CO₂ sequestration and the numbers are increasing over the time and the project can be driven in improving the effectiveness and safety of this approach.

1.6 Feasibility of Project within the Scope and Timeline

In the long run, this project is feasible to be done within the timeline drawn. In achieving the first objective, only several weeks required to be spent off on the reading and reviewing available data on the potential improvements and additional additives for the slurry. However, in time, a longer duration is needed to complete the second objective; to mix and evaluate the cement for deep CO₂ injection wells as there will be laboratory activities planned out and since trial-and-error method is to be implemented. Moreover, additives that are planned to be used are basically available in the university storages. Cement mixing and testing procedures will take about 12 weeks as there will be few lengthy procedures and need precision and accurateness data recording.

In addition, most of the equipments and tools needed in doing all the properties tests are available within the university facilities such as HPHT Consistometer, Ultrasonic Cement Analyzer, HPHT Cement Curing Chamber, Comprehensive Strength Tester and Stirrer Fluid Loss Tester. However, cured samples are to be sent to University Technology of Malaysia in Skudai, Johor to be tested on the CO₂ exposure using an Autoclave.

CHAPTER 2

THEORY & LITERATURE REVIEW

2.1 CO₂ properties

Deep injection CO₂ wells indirectly indicate that the bottomhole pressure and temperature will be greater than the normal wells but not exceeding the HPHT ranges (>10000 psi and >350°F). Therefore during the injection of CO₂ gas downhole, it will change its phase from gas to liquid before it becomes supercritical fluid. Table 1 shows the physical properties of CO₂ where it clearly expresses that CO₂ is in gas phase at ambient conditions with heavier density than air. In addition, thermodynamically, CO₂ appears as gas, liquid and solid at 75.1 psia and -69.9°F while no definite phase boundaries after CO₂ reaches 1071 psia and 87.9°F onwards.

Table 1: CO₂ Physical properties [12]

| English Unit | | Normal Boiling Point | | Gas Phase Properties | | | Liq Phase Properties | | Triple Point | | Critical Point | | |
|-----------------|-------|----------------------|-----------------------------|----------------------|---------------------------------|---------|----------------------|---------------------------------|--------------|------|----------------|------|---------|
| | | 1 atm | | @ 32°F & 1 atm | | | @ BP & 1 atm | | T | P | T | P | Density |
| Symbol | MW | T | Latent Heat of Vaporization | SG | Specific Heat (C _p) | Density | SG | Specific Heat (C _p) | T | P | T | P | Density |
| | | °F | BTU/lb | Air = 1 | BTU/lb °F | lb/cuft | Water = 1 | BTU/lb °F | °F | psia | °F | psia | lb/cuft |
| CO ₂ | 44.01 | -109.3 | 245.5 | 1.524 | 0.199 | 0.1234 | 1.18 | - | -69.9 | 75.1 | 87.9 | 1071 | 29.2 |

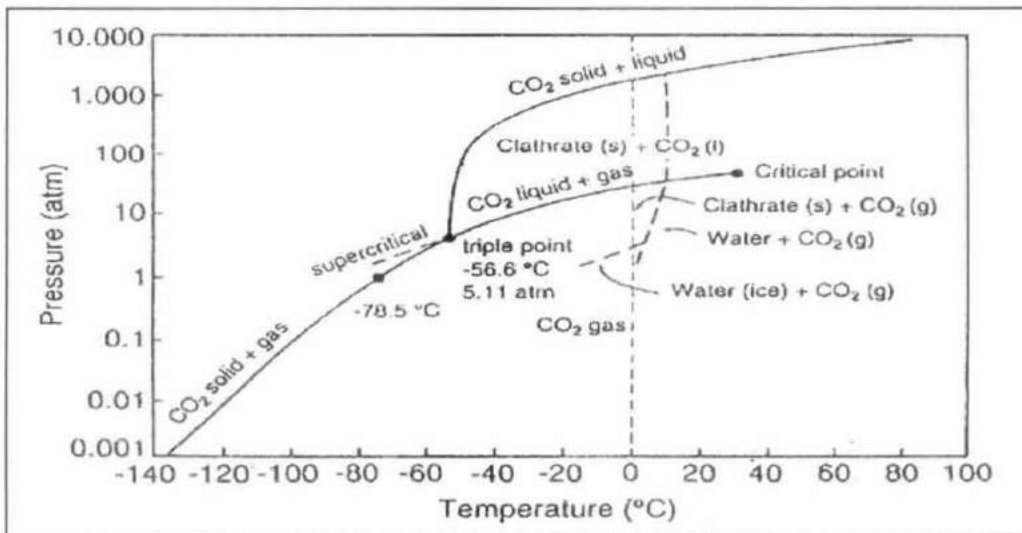


Figure 4: Pressure-Temperature phase diagram for CO₂ [13]

Figure 4 exhibits the phase diagram of CO₂ substance at varies temperature and pressure. Since the downhole condition of a deep well is exceeding 250°F (121°C) and 3000 psi (206.9 bar) which eventually greater than the critical points, this entails that CO₂ will be in supercritical fluid phase where it will expand like gas but with liquid density. However it is having lower toxicity and environment impact than the liquid phase.

2.2 Portland cement

Below are the equations to calculate the volume of cement required to be pump into the required hole with excess percentage.

$$C_h = \frac{ID_h^2}{1029.4} \quad (2.1.1)$$

$$C_{cd} = \frac{OD_{csg}^2}{1029.4} \quad (2.1.2)$$

$$V_{ann} = Z \times (C_h - C_{cd}) \quad (2.1.3)$$

$$C_{csg} = \frac{ID_{csg}^2}{1029.4} \quad (2.1.4)$$

$$V_{st} = C_{csg} \times L \quad (2.1.5)$$

$$V_t = V_{ann} + V_{st} \quad (2.1.6)$$

In Table 2, shown is the API standard 10A on the mixwater requirement for each type of standard Portland cement. Clearly stated in the specification by API 5.0 gallon of water should be added into a sack (94 lbs) of cement Class G in the industry practice which is represented as 0.44 water-cement ratio. In equivalent for laboratory purposes recommended by API [8] 264 mL of available mixwater is to mixed with 600 g of class G cement. However this is an average value which it is possible to be altered to suit different conditions.

Table 2: API cement with specific recommendation mixwater [10]

| 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 |

Based on API Recommended Practice 10B-2^[8], there are several important formulas that to be used in calculating the fluid loss and thickening time of a cement that means the requirement of ISO. For thickening time identification, equation 2.1.7 and 2.1.12 are applied;

$$t_d = \frac{V_p}{q} \quad (2.1.7)$$

$$R_{\Delta T} = \frac{T_{PBHC} - T_{SS}}{t_d} \quad (2.1.8)$$

$$R_{pu} = \frac{P_{BH} - P_s}{t_d} \quad (2.1.9)$$

$$t_a = \frac{V_a}{q} \quad (2.1.10)$$

$$R_{(\Delta T)a} = \frac{T_{TOC} - T_{PBHC}}{t_a} \quad (2.1.11)$$

$$R_{pd} = \frac{P_{BH} - P_{TOC}}{t_a} \quad (2.1.12)$$

And the 2 equations below represent the fluid loss where 2.1.13 is for no blow-out while 2.1.14 calculate the fluid loss in the case of blow-out during lab test

$$FL = 2 \cdot V_f \quad (2.1.13)$$

$$FL_{ISO} = V_f \frac{10.944}{\sqrt{t}} \quad (2.1.14)$$

Water-Cement ratio (W/C) is crucial because having too high ratio can cause weak cement setting and form an impermeable barrier while insufficient water content may result in increasing of cement density and viscosity which eventually will decrease the rheology/mobility.

Eventhough Class D, E and F cements are the most appropriate class of cement to be use in deep environment since it is also known as retarded cements and can be pump up to 16000 ft deep, for this project Class G will be the choice since it is more economically and most flexible type to be modified upon only using several additives as to compared to others. Moreover, in meeting the objective, new special additives will be introduced into the Portland cement explicitly Superplasticizer, Fillite and Silica Fume. Inert additives such as fly ash, silicate (sand and nitrogen (foamed cement) do not increase the water ratio and are assumed for this study to have a lesser impact on the deterioration of the cement in the presence of CO₂ (Nelson and Guillot, 2006a).

2.3 New additives

Blomberg claimed that mixture with microsilica and light weight materials may increase the permeability of the cement and ultimately reduce the penetration of fluid through it. To prove his theory he compared 2 type of slurry; Slurry A and slurry B and tested on the gas migration of both compositions. Table 3 represents the composition of slurry A where 15% and 4% BWOC of microsilica and fillite respectively were added as to compare to slurry B, composition in Table 4 where no accelerating additives were added (pure neat cement) in.

Table 3: Composition of sample A for gas migration test [3]

| Cement slurry A: | |
|--|------------------------|
| G-cement | 100.0 kg |
| Microsilica in the form of EMSAC 465T | 15.0 kg |
| Light weight aggregate: Fillite 0.7 | 4.0 kg |
| Sea water | 95.0 kg |
| Accelerator (Pozzolith 500A) | 0.6 L |
| Theoretical density for cement slurry A is | 1.55 g/cm ³ |

Table 4: Composition cement slurry B for gas migration test [3]

| Cement slurry B: | |
|--|------------------------|
| G-cement | 100.0 kg |
| Water glass (35 by weight dry material) | 15.0 kg |
| Sea water | 4.0 kg |
| Theoretical density for cement slurry B is | 1.55 g/cm ³ |

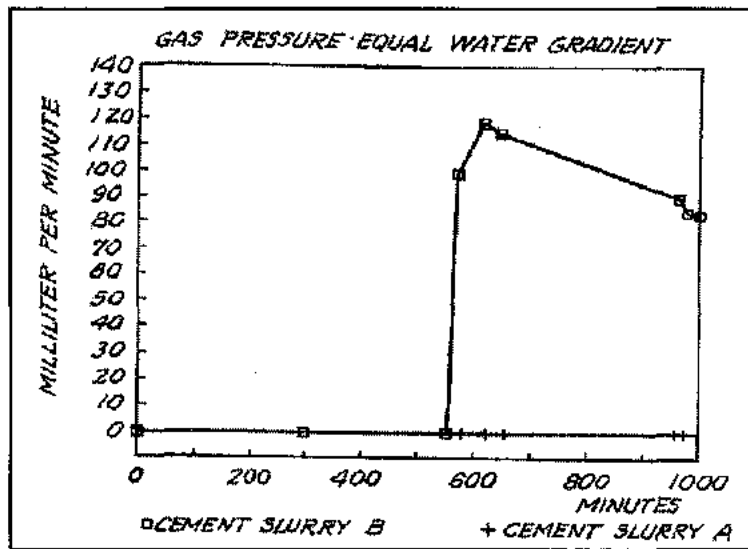


Figure 5: Gas migration/penetration through cement sheath plot [3]

The test succeeded when slurry A prove to impede the migration over a period of time and contradict the performance of slurry B which having an abrupt penetration after 550 minutes with 100 mm/min of penetration rate. The result was plotted and shown in figure 5.

2.3.1 Superplasticizer

Superplasticizer is a type of linear polymer that has the ability to reduce the water content in the cement mixture. It also can acts as a retarder to the composition which will be able to increase the thickening time by only increasing the composition percentile. The use of slag cement, silica fume and optimum superplasticizer content resulted in an increased compressive strength and good workability. [4]

2.3.2 Fillite

On the other hand, a slight percentage (about 1%) of fillite will be used to help in increase the volume with less water required. Fillite is a type of resins filler powder that is available in the university storage. Fillite is proven to be useful in helping by filling the hollow spaces within the mixture and prevent the gas migration through the sheath (Nils Blomberg – 1990). Fillite consists of spherical micro-fine particles and an adequate amount added can reduce the volume mixwater required with similar total volume of cement slurry.

2.3.3 Silica Fume

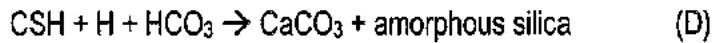
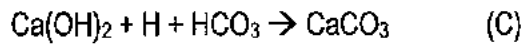
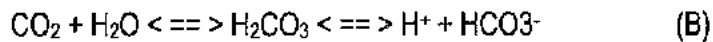
Meanwhile, silica fume is believed to have the potential in maintaining the strength of cement sheath during the CO₂ attack and reduce the density of the system (extender). This is a normal additive added for deep well composited by 85% - 95% of amorphous silicon dioxide (SiO₂). Having high specific surface area, this water wet matter has the ability to absorb the access water in cement slurry when the slurry is extended by water.

2.4 Cement Properties

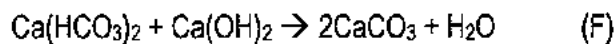
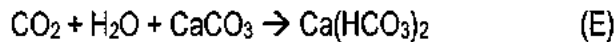
In making the best decision, the main 4 chemical, physical and mechanical properties that will be evaluated would be the CO₂ corrosion rate, compressive strength development, fluid loss rate and thickening time of those mixed compositions.

2.4.1 CO₂ Reaction

The cement should be able to partially resist the CO₂ attack to avoid any possible leakage. It is impossible to avoid any reaction with the acidic gas but having relatively low rate will expand the life time of the injection well longer. When calcium hydroxide (Ca(OH)₂) in contact with CO₂, the reaction will produce calcium carbonate (CaCO₃) and water (H₂O). Calcium carbonate formation will ultimately increase the permeability of the sheath and reaction 2.4.1A, 2.4.1B and 2.4.1C show the production of CaCO₃



Reactions C and D are contributing to the production of CaCO_3 which increase the permeability. However, this reaction does not stop; it continues to reaction E and F as below



With this, water produces from reaction f will dissolve more calcium bicarbonate and this unlimited water supply and Ca(OH)_2 downhole will leaves pores and corrode the cementing system downhole which reducing the strength of the cement in supporting the casing and preventing fluid to flow behind the casing (cementation leaking).

2.4.2 Fluid Loss

Fluid loss rate is the properties of the cement that determine the possible volume of liquid phase that will be loss from the pumping system and invade the formation. Having too high fluid loss will cause increase in cement viscosity and severe formation damage and gas migration within the sheath that will lead to the gas leakage through to the surface. As to comply the standard drawn by the American Institute of Petroleum (API), fluid loss of a cement system should be less than 70 cc/ 30 min.

2.4.3 Thickening Time

In the interim, an adequate thickening time is required in order to pump the system up to the desired settling depth. Thickening time is defined as the time required for the cement system to be harden (able to be pumped). Therefore, in ensuring the cement to harden at the specific designated depth/point, typical practice the thickening should be around 2 hours to avoid too long on wait of cement (WOC) during the development phase. 'Bottom hole circulating

temperature variation of up to 5°F will cause a considerable variation in thickening time' (Umeokafor, 2010)

2.4.4 Compressive Strength Development

Suitable compressive strength is critical in ensuring both the structural support for the casing and hydraulic as well as mechanical isolation of borehole intervals. This system designed shall resist against the in-situ pressure (pore pressure and fracture pressure), hydrostatic pressure exerted by the drilling fluid, thermal loads and periodic loads transferred from various operations that can cause cement hydration. There are 2 types of compressive strength; early age strength, (preparation phase to placement of the slurry) and long term strength (after several years of hydration). This project will only focuses in the early age strength where the development of the cement is evaluated to predict the possible gas migration through the solidification of the cement. To prevent gas migration during the settling, the transition time between liquid to solid phase shall be reduce where faster development of the strength is required.

2.5 Literature Review

For the cementing job in an acidic environment (CO₂), *'The high-alumina cement system holds a distinct advantage.... However present many challenges related to additive selection, prevention of contamination, material manufacture and quality control.'* [2] Here, Berge and Dew (2006) convinced that the new technology of high alumina cement has the potential in replacing Portland cement for CO₂ environment as a corrosion-resistance solution but require more researches, innovations and development of the new approach in the industry.

In oil well drilling industry class G and H well cements are known as basic well cements, because no additions other than calcium sulfate or water, or both, shall be inter-ground or blended with the clinker during manufacture of these well cement classes. Hence, with addition appropriate additives such as accelerators and retarders can change their setting time to cover a wide range of well depths, pressures and temperatures (Asadi, 1983, Nelson, 1999). Being widely used and better understanding on the properties of these Portland classes, cement class G is preferable in this project and alteration and modification will be imposed in improving the system that suits the conditions bottomhole.

When talking about the performance of silica fume and superplasticizer in a cement composition, P. Rathish Kumar claimed *'It is evident from the table that the addition of silica fume showed a gain in strength with age. An increase in the compressive strength of mortar was also obtained with a higher grade of cement. However, use of higher grade cements led to a decrease in the flow. Addition of an optimum dosage of superplasticizer to such a mix was, therefore, necessary to increase the flow of the mortar to be used for ferrocement works'*. This was claimed when he replaced cement with 10% silica fume, the compressive strength of the cement increases by over the timeline however it resulted in reducing of cement flow percentage.

'Recent laboratory work suggests, however, that cement with low free water ratios will not be susceptible to CO₂ attack due to the formation of an impermeable barrier on the cement sheath' [4]. From here, it is suggesting that new additives added into the mixture should also have the ability to absorb the free water volume to reduce the reaction between the cement and the acidic gas. This claim is seconding the result obtained by Blomberg when he obtained best result on gas migration/penetration from the cement slurry A (refer figure 5). Summarized outcome shown in Table 5.

Table 5: Comparison on the result for slurry A and B [3]

| | <u>CEMENT SLURRY</u> | |
|---|-----------------------------|------------|
| | A | B |
| Density, g/cm ³ | 1.53 | 1.53 |
| Apparent viscosity, cp | 24 | 20 |
| Plastic viscosity, cp | 18 | 16 |
| Yield point, lb/100 ft ² | 12 | 7 |
| Free water, volume % | 0 | 0.5 |
| <u>Setting time:</u> | | |
| Consistency 30BC | 3 h 15 min | 3 h 20 min |
| 100BC | 5 h 30 min | 5 h 20 min |
| Compressive strength after 24 hours at 30°C | 4.0 Mpa | 3.0 Mpa |

Using similar composition as in Table 3, sample A resulted in having 0% free water volume unlike sample B with 0.5% of free water volume, having 10 minutes extra on the thickening time and its compressive strength is higher than cement slurry B by 1.0 MPa. [3] However, no research has been done in determining the performance of the cement when it is

mix up with superplasticizer, fillite and silica fume as a system. Each materials are having different potentials and functions but with the same outcome to reduce the water content with better thickening time, fluid loss control, strength development and CO₂ resistance.

CHAPTER 3

METHODOLOGY

3.1 Research Methodology

Cement design and properties determination and testing will be done in the laboratory using specific tools, devices and equipments. The research methodology in determining the properties of the new cement composition based on API standard 10B[®] (NOTE: Neat cement is tested along with the new composition as a reference/datum result):

1. Prepare the mixture using Constant Speed Mixer
 - a. Weight dry materials and then blend thoroughly and uniformly prior to adding them to the mixing fluid.
 - b. Place the mixing container with the required mass of mix water and any liquid additives on the mixer base.
 - c. Turn on the motor and maintain at $4\ 000\ \text{r/min} \pm 200\ \text{r/min}$ ($66.7\ \text{r/s} \pm 3.3\ \text{r/s}$). (NOTE: If additives are present in the mix water, stir at the above rotational speed to thoroughly disperse them prior to the addition of cement. In certain cases, the order of addition of the additives to the mixing water can be critical. Document any special mixing procedures and mixing time).
 - d. Add the cement or cement/dry additive blend at a uniform rate, in not more than 15 s (if possible)
 - e. When all the dry materials have been added to the mixwater, place the cover on the mixing container and continue mixing at $12\ 000\ \text{r/min} \pm 500\ \text{r/min}$ ($200\ \text{r/s} \pm 8.3\ \text{r/s}$) for $35 \pm 1\ \text{s}$.
 - f. Measure and document the rotational speed under load
2. Test the well-simulation thickening time using HPHT consistometer
 - a. Assembly and filling the slurry container
 - i. clean and lubricate cup threads
 - ii. inspect diaphragm

- iii. assemble paddle shaft assembly and secure it in the cup sleeve with flange ring
 - iv. make sure the paddle turns freely
 - v. invert the slurry container assembly and fill to within 6 mm (1/4 in) of the top
 - vi. strike to remove air
 - vii. screw in base plate and make sure slurry is extruded through the centre hole
 - viii. screw centre plug (pivot bearing) into the container
 - ix. wipe all cement from the outer surfaces
 - x. recheck the paddle to make sure it turns smoothly
 - xi. load the slurry container assembly into the consistometer
- b. Initiate the test
- i. Place the filled slurry container on the drive table in the pressure vessel
 - ii. Start rotation of the slurry container and secure the potentiometer mechanism so as to engage the paddle shaft drive bar.
 - iii. Begin filling the vessel with oil. (At this point, the paddle shaft shall not be rotating).
 - iv. Securely close the head assembly of the pressure vessel, insert the temperature-sensing device through the hole in head assembly and partially engage the threads.
 - v. After the pressure vessel is completely filled with oil, tighten the threads of the temperature-sensing device.
 - vi. Begin operating the apparatus 5 min \pm 15 s after cessation of mixing of the slurry
- c. Control temperature and pressure
- i. During the test period, increase the temperature and pressure of the cement slurry in the slurry container in accordance with the appropriate well-simulation test schedule
3. Test the fluid loss at Low Temperature Low Pressure
- a. Prepare and complete the filtration unit (filter net, filter paper, O-rings etc)
 - b. Prepare and mix the cement
 - c. Pour the slurry into a clean and dry filtration unit

- d. Complete the assembly connect the gas line together
 - e. Apply and maintain 100 psi to the cell
 - f. Once the pressure is applied, time the test
 - g. Record the volume of collected water at 0.5 min, 1 min, 2 min, 5 min, 10 min, 15 min, 25 min and 30 min
4. Test the development of compressive strength using Ultrasonic Cement Analyzer
- a. Prepare and lubricate the test cell
 - b. Check on the heating element at the base of the cell and lubricate it
 - c. Mix the cement and pour it into the cell using the UCA indicator up to the wet fill level (figure 6)
 - d. Slowly added water into the cell up to the water fill level using rod to avoid water mixing with the cement system.
 - e. Close the cell tightly and place it into the analyzer cell.
 - f. Connect the 'J' tube tightly onto the cell and thermocouple hand-tighten into the other inlet.
 - g. Open the water supply valve and once fully filled, tighten the thermocouple using 5/8" wrench
 - h. Set the temperature and the shutdown time (24 hours) and start the test.
 - i. Switch the heater and the pump on. Meanwhile, adjust the pressure in the cell using the pressure adjuster knob till the desired pressure and maintain it.



Figure 6: UCA filling indicator

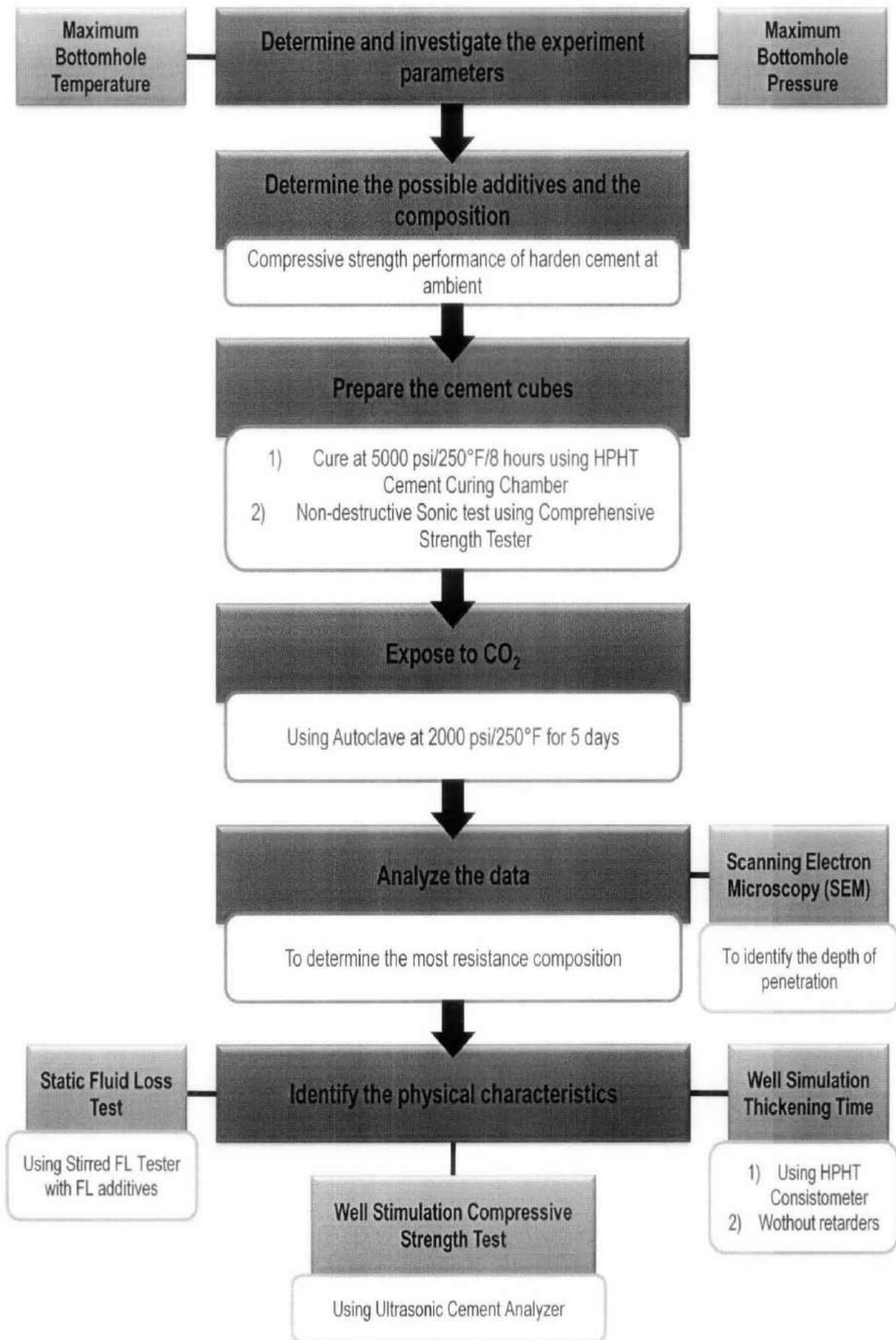





Figure 7: Overall flow methodology

3.2 Tools & Equipment Needed – Testing Procedure

This section will explain further on the functions for all available equipments that will be used throughout the research mainly on preparing the laboratory results and data. Generally, there will be 6 main facilities available to assist me for the preparation, they are as explained:

| Equipment | Function |
|--|--|
| <p>(1) Constant Speed Mixer</p>  <p>Figure 8: Constant Speed Mixer [8]</p> | <p>To mix the cement slurries with constant speed regardless of the torque exerted on the mixing blade</p> |
| <p>(2) HPHT Consistometer</p>  <p>Figure 9: HPHT Consistometer [8]</p> | <p>To evaluate the thickening time or pumping time of cement slurry under simulated downhole conditions of temperature and pressure</p> |
| <p>(3) Ultrasonic Cement Analyzer</p>  <p>Figure 10: Ultrasonic Cement Analyzer [8]</p> | <p>To determine the compression strength on the cement mixture over the function of time by constructing a continuous non-destructive method</p> |

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 Data Gathering

Most researches that test these 3 additives were done separately into different cement system and that resulted in one specific study of an additive. However, this paper will discuss further on the performance of the system when having all 3 additives into a system and how it can improve the performance that will compensate the deep CO₂ injection well in the field/real practice.

For the purposes of testing and proving the significance of adding new additives in a Portland cement, 3 additives are to be added in explicitly ELKEM MICROSILICA, TRELLENERG FILLITE and SIKA VISCOCRETE. Each of these additives is having different functions but with the same mission to avoid leakage, increase injectivity performance and the lifetime of the well. Table 6 shows the compositions and detail functions of each additive. Silica fume and Fillite are common type of replacers in concrete and have the potential of strengthen the concrete by replacing a percentile of cement. Meanwhile superplasticizer works in reducing the free water volume in the cement by absorbing the excessive volume.

Table 6: Roles of Additional Materials [1, 3, 4, 5]

| ADDITIONAL MATERIALS | COMPOSITIONS | DETAIL FUNCTIONS |
|---------------------------------------|---|--|
| Elkem Microsilica (silica fume) | SiO ₂ = 90.3%, CaO = 1.25% | Strength retrogression has been found in neat cement more than 110 °C. As such, silica was added to increase the strength of neat cement. |
| Trelleberg Fillite (fillite) | SiO ₂ = 51.5%, Al ₂ O ₃ = 43.4%, CaO = 1.53% | This inert material gave a less reaction of cement to CO ₂ and beneficial for the light weight cement composition. It can be used as extender in cement. |
| Sika Viscocrete (superplasticizer) | S = 40%, O = 60% | High water reducer and can be acted as retarder for high temperature well. Reduce the amount of water used in cement and increase permeability and porosity of cement. Thus, possibly enhanced the cement resistance towards CO ₂ attack. |

4.1.1 CO₂ Reaction

Having too high amount of additives will incur extra cost on the operation. With that reason, minimum percentages of additives are used and the performances are tested and analyzed. Along with that only 1% of fillite and 0.5% of superplasticizer are added while silica fume varies from 0% to 35% with lower range of silica fume at 0.44 W/C and higher range with 0.5 W/C. Table 7 summarizes the composition and the depth of penetration after 3 days of CO₂ exposure.

Table 7: Composition for CO₂ reaction test

| Cement Additives | Composition | | | | |
|---------------------------|-------------|------|-------|-------|-------|
| | 1 | 2 | 3 | 4 | 5 |
| SF (%BWOC) | 35 | 25 | 15 | 5 | - |
| F (%BWOC) | 1 | 1 | 1 | 1 | 1 |
| SP (%BWOC) | 0.5 | 0.5 | 0.5 | 0.5 | 0.5 |
| W / (C + SF) | 0.5 | 0.5 | 0.5 | 0.44 | 0.44 |
| Depth of penetration (μm) | 1843 | 1350 | 165.1 | 131.4 | 146.1 |

As shown in Table 7, composition 1 and 2 are resulted in even deeper penetration than the neat cement performance (386μm) and therefore the composition were eliminated from the project. Generally, carbonation reduces by reducing the water ratio and additional of silica fume. However, water ratio implies higher impact on the reaction. Figure 11, 12 and 13 shows the Scanning Electron Microscopy (SEM) results for composition 3, 4, and 5 respectively. The depth of penetration indicates the carbonation reaction when CO₂ effectively reacted towards the cement sheath which clearly seen that composition 4 is having best resistance.

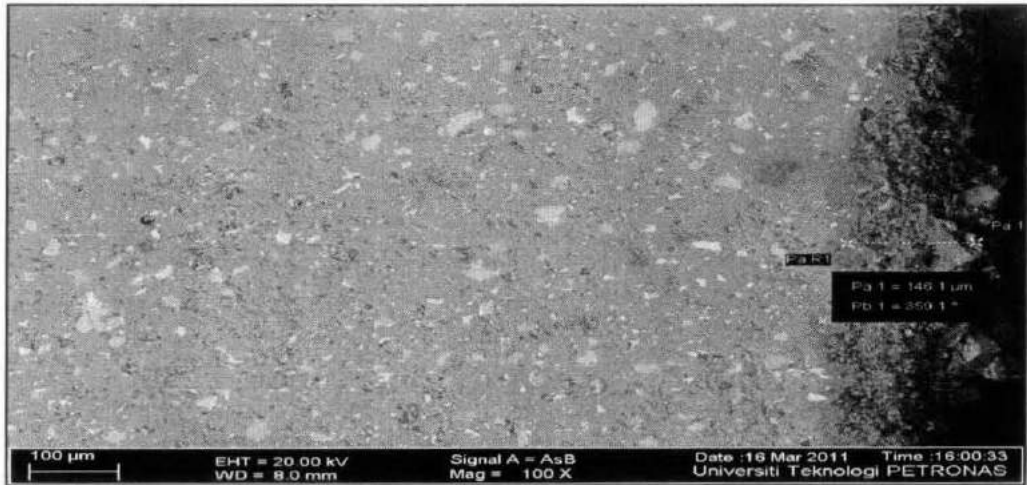


Figure 11: Microscopic view for composition 3

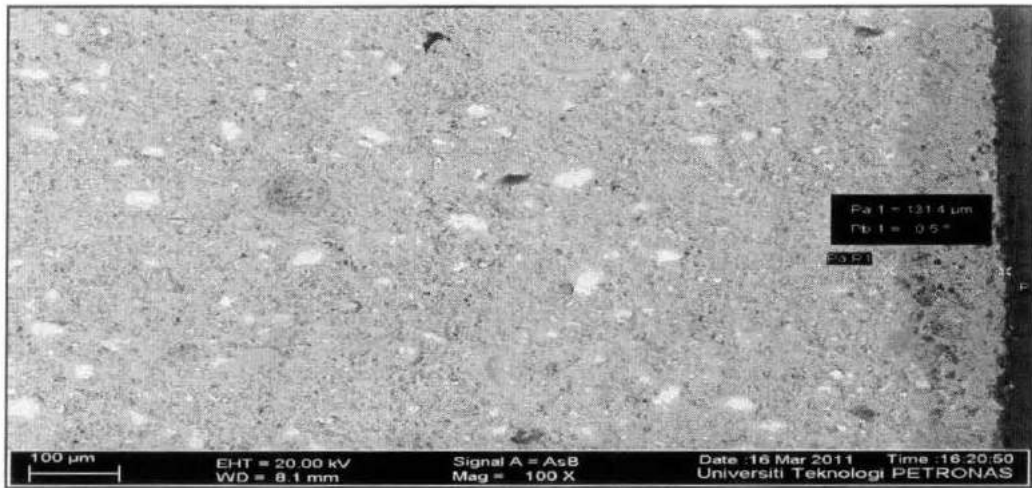


Figure 12: Microscopic view for composition 4

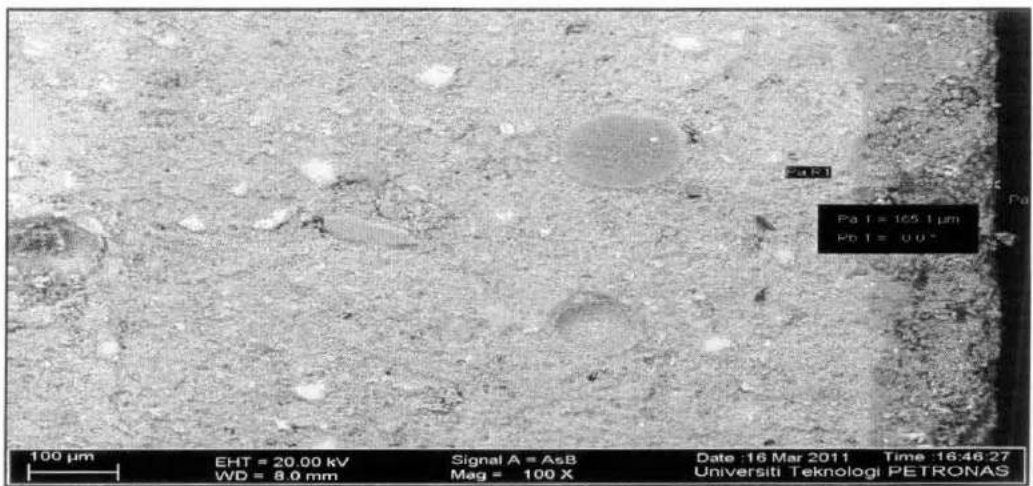


Figure 13: Microscopic view for composition 5

From this reaction result, the best composition would be the fourth composition which having the less depth of penetration. From that, this exact composition is used to determine the compressive strength, fluid loss and thickening time. Table 8 shows the weightage of each component in the system.

Table 8: Cement Composition for physical tests

| Materials | Percentage | Weight |
|------------------------------------|------------|---------|
| Cement Class G | | 600 g |
| Silica Fume (Elkem Microsilica) | 5% BWOC | 30 g |
| Fillite (Trelenberg Fillite) | 1% BWOC | 6 g |
| Superplasticizer (Sika Viscocrete) | 0.5% BWOC | 3 g |
| Water ratio | 0.44 | 277.2 g |

This composition is the basic composition uses in determining the other physical properties namely the compressive strength development, fluid loss rate and thickening time.

4.1.2 Compressive Strength Development

Using the Ultrasonic Cement Analyzer (UCA), the development of the system was tested for 4 different conditions for only 24 hours of duration as shown in Table 9. This test was conducted in examining the potential of the cement to have sufficient time on WOC and transition time that is crucial is supporting the casing and hinders the gas migration during the cement hydration.

Table 9: Compressive Strength testing conditions/cases

| Pressure | Temperature | Time (HH:MM) | | |
|----------|-------------|--------------|---------|--------|
| | | 50 psi | 500 psi | Max CS |
| 3500 | 250 | 02:05 | 03:22 | >24:00 |
| 4000 | 300 | 01:58 | 02:45 | 16:01 |
| 4250 | 325 | 01:42 | 02:33 | 14:10 |
| 4500 | 350 | 01:34 | 02:13 | 10:02 |

Overall, the new system performed better than the neat cement where the compressive strengths are happen to be around 2 hours in reaching the gel strength (50 psi) and less than 3.5 hours of Waiting on Cement (WOC) time which signify the time for cement to gain minimum compressive strength of 500 psi for resisting the shocks caused by drilling operation at the later stage [5]. For the first condition at 3500 psi and 250°F the maximum strength was 2880 psi at 24:00 when the test auto-stop and the strength is still increasing after 24 hours. Meanwhile the maximum strengths at 4000 psi (250°F), 4250 psi (250°F) and 4500 psi (250°F) are 2500, 2100 and 1900 psi respectively.

4.1.3 Fluid Loss Rate

Different approach was implemented to test the fluid loss of the new mixture. The mission was to prove that the fluid loss rate is easier to be controlled as to compare to the neat cement. During the lab work, fluid loss additive used was the FL-45 LS and added into a neat cement mixture and the new composition. The test was done at low temperature (room temperature) and low pressure of 100 psi. The volume of fluid collected for both slurries are recorded and figure 14 represents the improvement of the new composition in term of the loss rate.

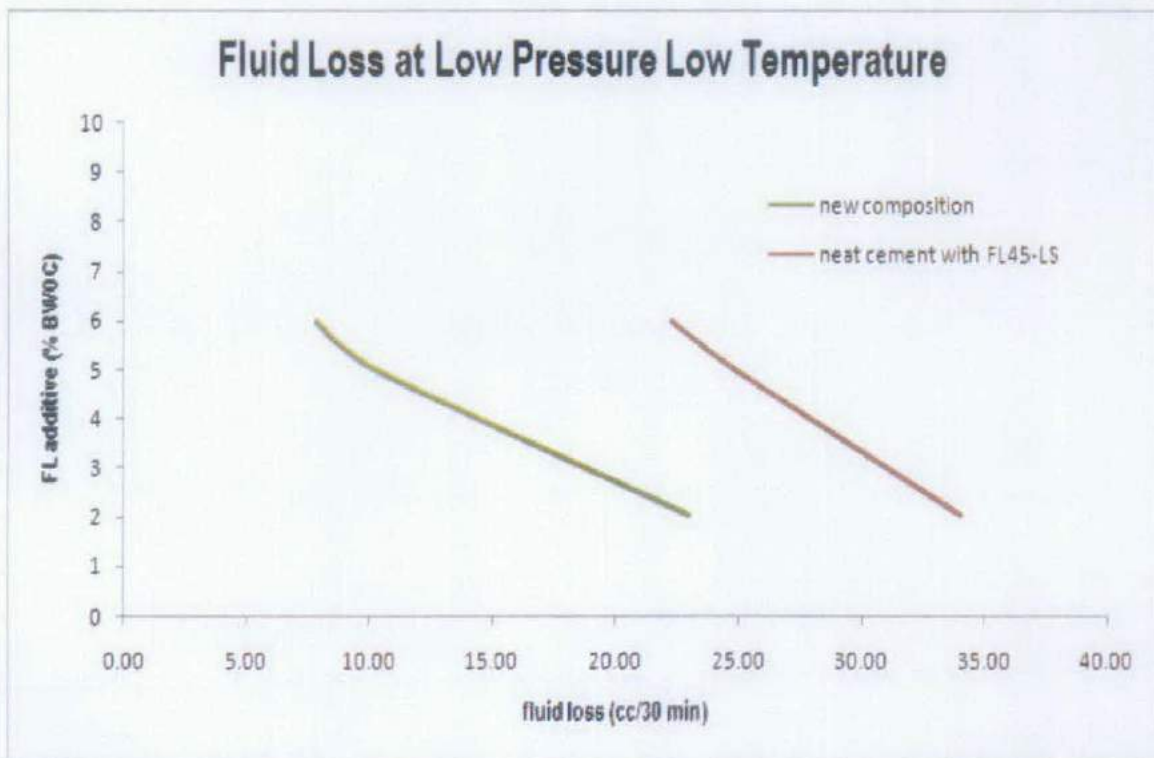


Figure 14: Comparison performance between new cement and neat cement with FL additive

In comparing the new cement performance and normal neat cement with common fluid loss control additives as to produce Figure 14, the new composition and neat were added with similar amount of additive and proven to be superior in controlling the loss. Protecting the fluid from leaving the system abruptly can allow the cement to hydrate in stable condition and cause less formation damage and better cementing job where less bubble pores left during the hardening duration of the cement.

4.1.4 Thickening Time

For consistometer testing as resulted in Figure 15, only a test were conducted at 4500 psi and 350°F for the new composition without any retarder was added in. This is to make the result as the datum and reference point for further test at higher or lower condition and with or without retarders. In addition, no retarders were added into the system to allow a raw analyze on the potential of the additives in allowing deeper pumping and longer distance before the settlement. However, the pressure rate up of this test was very high at 565.2 psi/min while temperature gradient was at 7.8°F/min (1.94°F/100 ft).

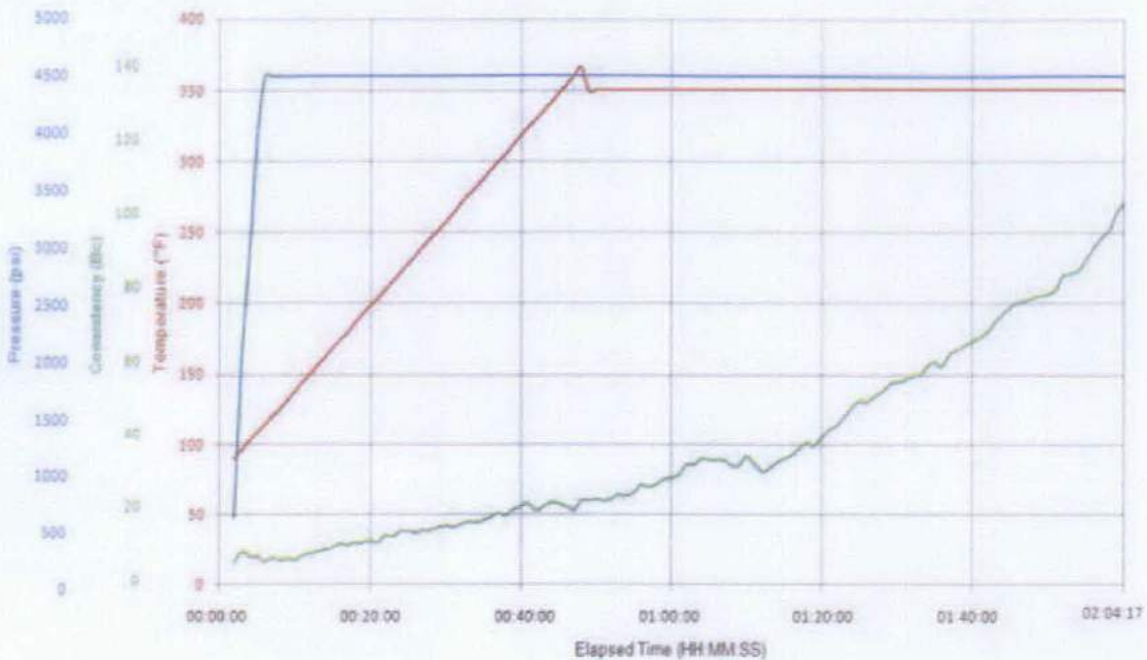


Figure 15: Consistometer result for thickening time at 4500 psi (350°F) with 0% of retarder

It is proven that the system allow deeper cementation as it required around 2 hours to harder without any retarders. Unlike neat cement which can only sustain less than 1 hour without

any retarder added. This means the system can work longer if retarders are added in and lower condition of bottomhole.

4.2 Data Analyzing

4.2.1 CO₂ Reaction

During the hydration of the cement, an extensive amount of carbon hydroxide and water are being produced and the chain continues unlimitedly as long as water and Ca(OH)₂ are massively generated bottomhole. Therefore, by reducing the water/cement ratio (WCR) of the system, it will contribute to limited reaction between the water and CO₂ which will lead to restricted generation of carbonic acid [HCO₃]. This is proven when the test results show that 0.44 water ratio composition is having less penetration than those with 0.5 WCR.

However, in reaction 2.4.1A, even with low water ratio, water can still be created downhole when CO₂ is in contact with Ca(OH)₂ and this contributes to higher penetration of composition 5 as compared to composition 4 that was considered 5% of silica fume. Mueller (1991)^[6] claimed that silica fume has the ability to reduce the Ca(OH)₂ content and neutralize the excess of Ca(OH)₂ when it replaces a certain portion of the cement.

Besides that, superplasticizer acted as the high range water reducing admixture when it helps to reduce the water content of the cement. Even though water can be generated downhole but from the test run, water ratio contributes severe impacts as water is a common product from any reaction between acid and it is the main contributor in producing corrosive carbonic acid downhole.

4.2.2 Compressive Strength Development

Based on Mueller's study, he claimed that silica fume will increase the strength development of cement due to the increased rate of hydration and increased pozzolanic reacting of silica fume cement. The rate of hydration increases because silica fume has a high surface area and eventually will absorb the excessive water and this leads to faster hydration when there is less free water in the system.

In addition, Shadizadeh (2010) supported the idea by indicating that silica fume has fine particles and this made it a good micro-filler between the cement particles to

increase the strength of the cement. Other than that, he stated that silica fume is a reactive solid to pozzolanic reaction and lead to shorter transition time and less time for gas migration. During the performance study between silica cement and fly ash cement, Mehta & Gjørv found out that silica is giving higher compressive strength fly ash. Nevertheless, when too high silica is added, it will abruptly reduce the free water within the system but eventually this will bring to the increase of viscosity of the system. This caused the best composition for the project is at the most optimum amount of silica fume (5% BWOC).

4.2.3 Fluid Loss Rate

Lower water ratio means lower free water amount. As a result, lower fluid loss rate is expected from the composition and the laboratory test confirmed on the idea. During the test, it is clearly seen the system formed an effective permeable cement cake layer at the filtration unit and left plenty water in the cement. Having particle sizes of 0.15 – 0.3 μm which is 100 times finer than cement particles, silica fume acted as particulate agents in the filter cake and abruptly reduce the fluid loss from the system into the permeable formation. These fine particles will block and lodge between those cement particles and lump narrow channels of the cement and decrease the permeability of the cake. This answers why during the first 10 minutes, the fluid loss at higher rate but eventually it slows down immediately when the filter cake allow harden at the bottom of the unit.

Silica fume and superplasticizer are also in the system to reduce the free water by absorbing out that excessive amount since these additives are having water wet of sufficient high specific surface area. 'Increase of water to cement ratio (WCR) of cement slurry causes the cement permeability to increase' (Larson – 1963) and is reasonable when water has the potential of creating effective channels within the cement during the hydration of the cement particularly when water tend to leave the system into more permeable formation of dehydrated out due to changes of temperature.

4.2.4 Thickening Time

In his writing, Shadizadeh indicated that thickening time of high WCR slurry is longer but lead to lower compressive strength of the cement sheath. This is reasonable when more water in the system, more time is needed to fully dehydrate the water out from the system.

Therefore silica fume in this test caused faster settling of the system and this is contradicted with the result obtained from the lab. This had had to relate with the existence of superplasticizer and fillite in the system to help to delay the time for deeper and longer pumping distance. Superplasticizer specifically has been classified by ASTM¹⁹ as water reducers and retarders in cement class G where it had the tendency to control the water ratio but produce a workability composition. In addition, fillite is consisted by spherical particles and this assist in reducing the internal friction during the pumping and allowing additional time and distance for pumping.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

In a nut shell, it can be concluded that,

1. The most suitable composition of silica fume, fillite and superplasticizer for deep CO₂ injection well is 5%, 1% and 0.5% respectively with only 0.44 water-cement ratio
2. The amendment on the composition managed to:
 - a. Increase the resistance towards CO₂ attack by up to 65%.
 - b. Improve the cement strength development at higher conditions.
 - c. Allowing deeper and longer pumping time and distance.
 - d. Improve the fluid loss rate from 35% to 65% than neat cement.

5.2 Recommendations

After completing these projects, there are several other further studies that will help the idea to be strongly justified and explained for industry applications. These are several improvements and additional researches suggested:

1. Test on the permeability before and after the CO₂ attack to check on the permeability of the sheath in sealing the formation fluid from channeling through behind the casing from bottomhole to surface.
2. Increase the settings to HPHT conditions to study on the reliability of those additives and cement class G in more extreme conditions.
3. Perform XRD on each sample after physical tests to better understand in detail and more microscopic view on the affect of those additives to the cement.
4. Test on the brittleness of the cement sheath in estimating the shattering characteristic of the cement during perforation.

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APPENDIX 1

PROJECT GANTT CHART

| Activities | 2010 | | | | | 2011 | | | | |
|--|------|---|---|---|---|------|---|---|---|---|
| | A | S | O | N | D | J | F | M | A | M |
| Read and review some literature materials on the alternatives for CO2 environment cementing system | | ■ | ■ | ■ | | | | | | |
| Determine the composition of suitable cement and its additives | | | | | ■ | ■ | | | | |
| Mix the cement slurry | | | | | ■ | ■ | | | | |
| Cure the mixtures at 8000 psi and 250°F | | | | | ■ | ■ | | | | |
| Evaluate the compressive strength of the cured cement | | | | | ■ | ■ | ■ | | | |
| Determine the chemical resistance of the cement towards high concentration of CO ₂ flow | | | | | ■ | ■ | ■ | | | |
| Determine the cement strength development at varies condition | | | | | | ■ | ■ | ■ | | |
| Determine the thickening time at test maximum bottomhole condition | | | | | | ■ | ■ | ■ | | |
| Determine the fluid loss rate at low pressure and low temperature | | | | | | | | ■ | ■ | |
| Resulting and Documenting | | | | | | | | | | ■ |

APPENDIX 2

PROJECT KEY MILESTONES

| Milestone | 2010 | | | | | 2011 | | | | |
|----------------------------------|------|---|---|---|---|------|---|---|---|---|
| | A | S | O | N | D | J | F | M | A | M |
| Completion on materials reading | | | | ■ | | | | | | |
| Completion of cement compositing | | | | | | ■ | | | | |
| Completion of cement evaluations | | | | | | | | | ■ | |
| Completion of research | | | | | | | | | | ■ |

APPENDIX 3
THE ADDITIVES



Elkem Microsilica (Silica Fume)



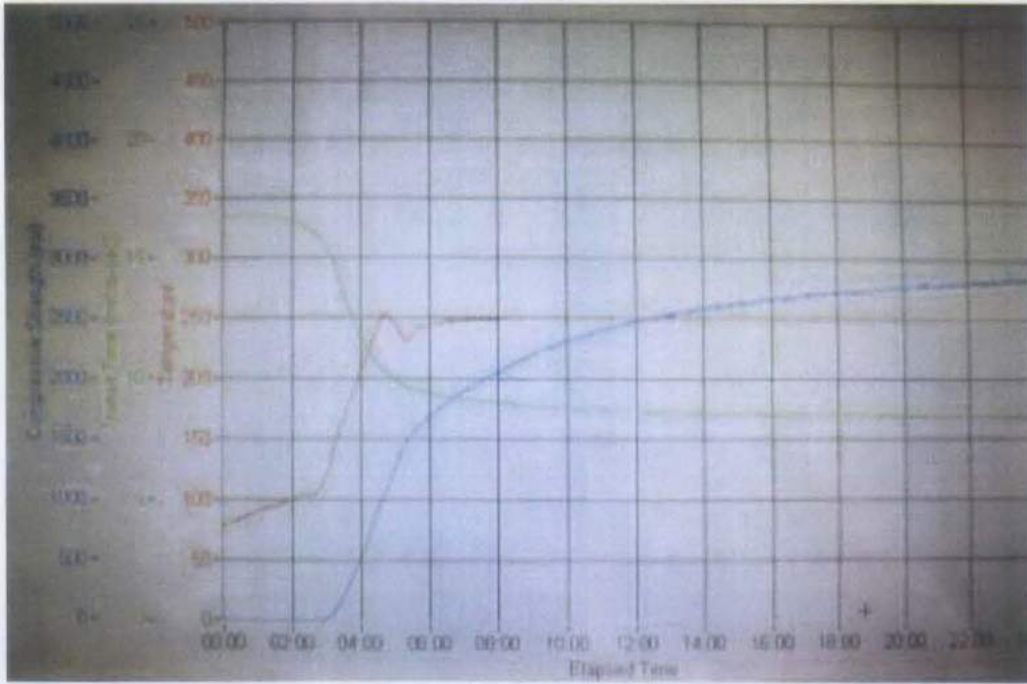
Trellenberg Fillite (fillite)



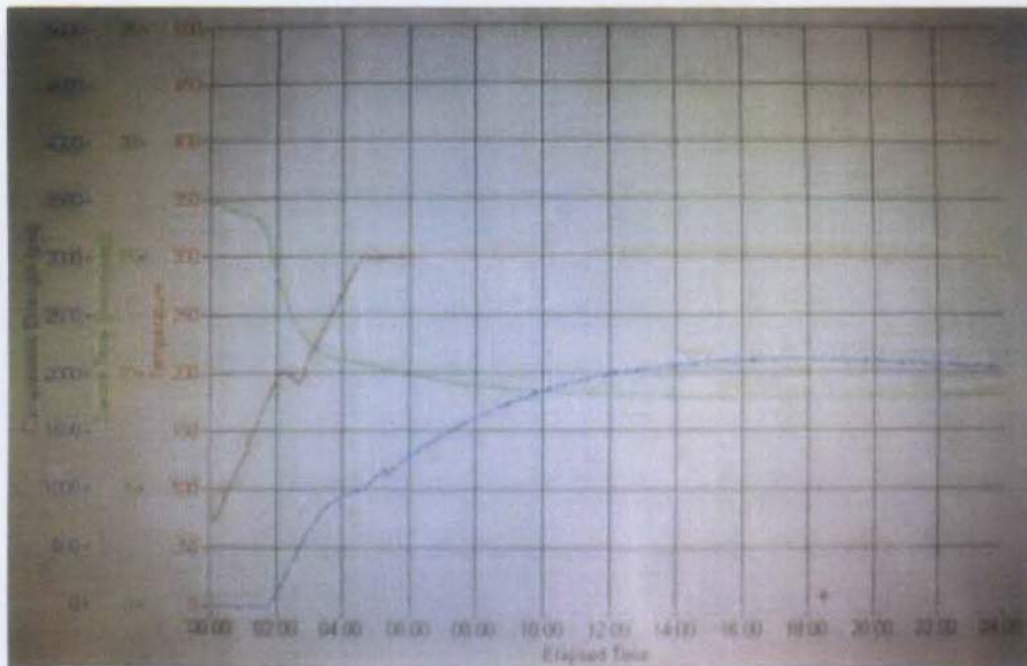
Sika Viscocrete (Superplasticizer)

APPENDIX 4

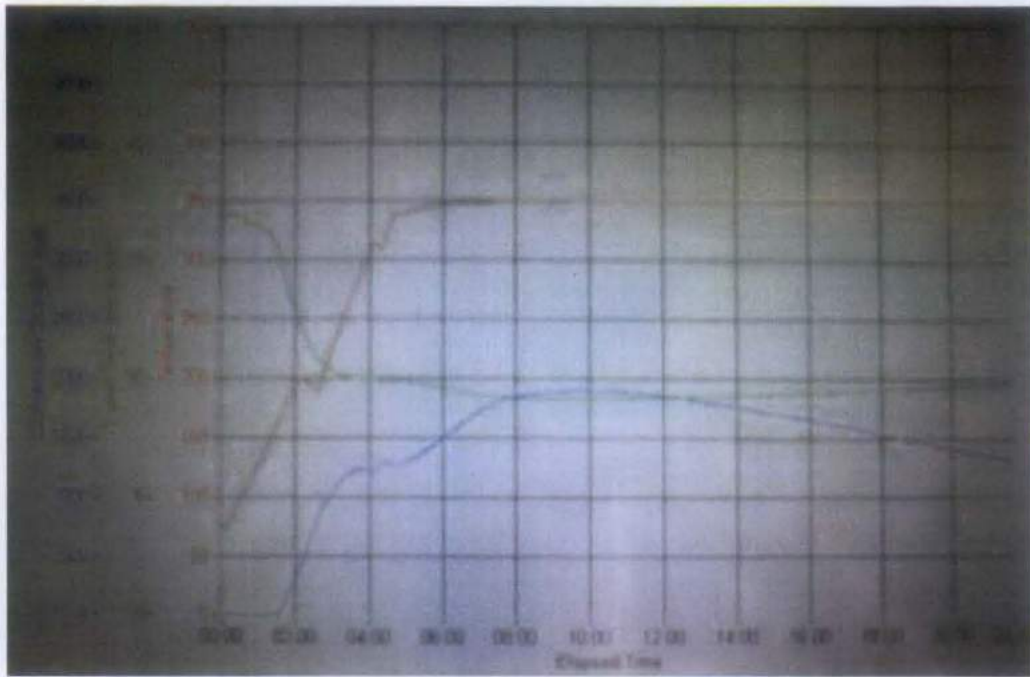
ULTRASONIC CEMENT ANALYZER OUTPUTS



Strength development at 3500 psi (250°F)



Strength development at 4000 psi (300°F)



Strength development at 4500 psi (350°F)

APPENDIX 5

FLUID LOSS WATER VOLUME

| 2% additives | |
|---------------------|-----------------------------|
| Time (min) | Water collected (mL) |
| 0.5 | 0.5 |
| 1 | 1 |
| 2 | 1.8 |
| 5 | 4.2 |
| 7.5 | 6.1 |
| 10 | 8.3 |
| 15 | 12.5 |
| 25 | 19.6 |
| 30 | 23 |

| 5% additives | |
|---------------------|-----------------------------|
| Time (min) | Water collected (mL) |
| 0.5 | 0 |
| 1 | 0.5 |
| 2 | 1.2 |
| 5 | 3.8 |
| 7.5 | 5.2 |
| 10 | 6.4 |
| 15 | 7.1 |
| 25 | 9.6 |
| 30 | 10.2 |

| 6% additives | |
|---------------------|-----------------------------|
| Time (min) | Water collected (mL) |
| 0.5 | 0 |
| 1 | 0 |
| 2 | 0.8 |
| 5 | 3.5 |
| 7.5 | 4.1 |
| 10 | 4.7 |
| 15 | 5.4 |
| 25 | 7 |
| 30 | 7.8 |