

GEOPOLYMER CEMENT FOR OIL WELL WITH IMPROVED PROPERTIES

By

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CERTIFICATION OF APPROVAL

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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.

Wan Ahmad Fahmi Azrin Che Mohd

ABSTRACT

With the increasing awareness of global warming, geopolymers have been identified as one of the methods in reducing the emission of CO₂ during oil well cementing operation. However, it is important that geopolymer cement can meet the specific requirement of oil well condition in order to be the substitute of current conventional cement system. The use of geopolymer in cement system is a new technology that yet needs proper study to yield better advantages of it.

In this research, the main objective was to observe properties are thickening time, fluid loss and compressive strength. In the early stage, literature review on previous research showed utilizing geopolymer in cement composition will significantly reduce CO₂ emission and enhanced properties characteristic as well. Detailed study on geopolymer materials, conventional cement, and additives was carried out.

As the conclusion, from the obtained results geopolymer cement showed better properties compared to conventional cement.

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Cementing operation consists of a very important work for the following drilling and completion operations of oil well and has a great impact on the productivity of the oil well. The challenges in oil well cementing are to design cement slurries that meet the specific requirement of well condition [1]. Current conventional well cementing materials are Portland cements with the use of additives to manipulate its properties [2].

Unfortunately, according to the government of Canada greenhouse report, conventional Portland cements one of the major greenhouse gases producers [3]. With the increasing pressure to adopt green practices in cementing activity, it is important to develop environmental friendly cement slurry which can be used as a substitute. Studies have been done on using geopolymer material in cement slurry system and it is proven to produce less greenhouse gases [3-6].

In order to apply the use of geopolymer in oil well cementing, detail study regarding the properties of geopolymer cement system is crucial. In recent study [3], the geopolymer cement composition showed better performance of properties such as compressive strength, pumping time, fluid loss control and viscosity. To further enhance the understanding, this research will focus on the properties of geopolymer cement compositions and determine the best composition.

This research is done experimentally consisting standard weight cement slurry for simulated oil well conditions. The properties that will be measured are thickening time, fluid loss and compressive strength development.

1.2 Problem Statement

1.2.1 Problem Identification

Portland cement is a major construction material used worldwide. Unfortunately, the production of Portland cement releases large amounts of CO₂ into the atmosphere making a major contribution to the greenhouse effect and the global warming of the planet. Portland cement production is estimated to contribute around 7% of global CO₂ emissions [5]. To reduce greenhouse gas emissions, efforts are needed to develop environmentally friendly materials to replace the use of Portland cement.

One such alternative is geopolymer cement, unlike Portland cement, the chemical reactions that form the geopolymer cement do not require high temperatures for processing which give off carbon dioxide. The geopolymer cement manufacture is estimated to reduce CO₂ emission by 22% to 72% (depending on materials used) [5].

1.2.2 Significant of the Project

In order to replace the use of conventional cement material with geopolymer cement in oil well cementing, it is crucial to ensure its properties can meet the design specification requirement. Thickening time, fluid loss and compressive strength development are some of the vital properties for a cement slurry system [2]. This is because an adequate thickening time is required to enable cement slurry to be pumped down the wellbore, fluid loss is the unwanted migration of the liquid part of the cement slurry into a formation, and cement compressive strength is required to protect casing from formation pressure [2,6].

1.3 Objective

Study the properties of geopolymer cement on thickening time, fluid loss, and compressive strength.

1.4 Scope of Study

The scope of study is mainly on designing geopolymer cement compositions, preparing conventional cement composition and testing in accordance to the American Petroleum Institute API-RP-10B. The obtained results will be compared in terms of the properties

(thickening time, fluid loss and compressive strength) with conventional cement slurries. The study will comprise standard weight cement slurry.

1.5 The Relevancy of the Project

Current trends in oil well cementing are approaching the application of geopolymer in designing the cement slurry and as the use of geopolymer in oil well cementing is still new, a lot of studies need to be carried out in order to gain better knowledge in geopolymer technology. The obtained results from this project are also applicable for real oil well as the procedures comply with American Petroleum Institute specifications.

1.6 Feasibility of the Project

This project is encompassing research and also laboratory work. Most of equipment and material are already available at Drilling Fluid Laboratory which is under Geosciences & Petroleum Engineering Department and geopolymer can be produce with the collaboration from Chemical Engineering Department. This project can be done within 8 months given that everything goes fine, the objectives can be achieved if the procedures are closely followed.

CHAPTER 2

THEORY AND LITERATURE REVIEW

2.1 Theory

2.1.1 Geopolymers

Alumino-Silicates based geopolymer

The reaction of a solid aluminosilicate with a highly concentrated aqueous alkali hydroxide or silicate solution produces a synthetic alkali aluminosilicate material generically called a ‘geopolymer’, but probably more appropriately referred to as an example of what is more broadly termed an ‘inorganic polymer’ [10].

Geopolymers based on alumino-silicates are generally designated as poly(sialate), which is an abbreviation for poly(silicon-oxo-aluminate) or $(-\text{Si-O-Al-O-})_n$ (with n being the degree of polymerization)[10]. The sialate network consists of SiO_4 and AlO_4 tetrahedra linked alternately by sharing all the oxygens, with Al^{3+} and Si^{4+} in IV-fold coordination with oxygen and range from amorphous to semi-crystalline. Positive ions must be present in the framework cavities to balance the charge of Al^{3+} in IV-fold coordination [10]. The amorphous to semi-crystalline three dimensional silico-aluminate structures were christened of the types show in Table 2.1.

Table 2.1: Geopolymers chemical designation [10]

Si/Al ratio	Designation	Structure	Abbreviations
1	Poly(sialate)	Mn(-Si-O-Al-O-)n	(M)-PS
2	Poly (sialate-siloxo)	Mn(-Si-O-Al-O-Si-O)n	(M)-PSS
3	Poly (sialate-disiloxo)	Mn(-Si-O-Al-O-Si-O-Si-O-)n	(M)-PSDS

M is a cation such as potassium, sodium or calcium, and n is a degree of polymerization.

For Si:Al>>3:1, the polymeric structure results from the cross linking of polysilicate chains or sheets with a sialate link (-Si-O-Al-O-) (2D or 3D cross-link).

Phosphate-based geopolymer

Phosphate ceramics are synthesized at room temperature and they set rapidly like conventional polymers. They contain naturally occurring mineral phases, notably apatite. They represent another variety of mineral geopolymer, where Si is totally or partially replaced by P. They are formed by an acid-base reaction between a metal oxide and an acid phosphate. Virtually any divalent or trivalent oxide that is sparingly soluble may be used to form these phosphate geopolymers [10]. They have found a wide range of applications such as dental cements, construction materials, oil well cements, and hazardous and radioactive waste stabilization. The main difference between the silicate based geopolymers and phosphate geopolymers, however, is their syntheses. Poly(sialate) geopolymers and their derivatives are synthesized in alkaline environment, but phosphate geopolymers are fabricated by acid-base reactions.

A very wide range of phosphate geopolymers may be synthesized by acid-base reaction between an inorganic oxide (preferably that of divalent and trivalent metals) and an acid phosphate [10]. The reaction product is generally a poly(hydrophosphate) or an anhydrous poly(phosphate) that consolidates into a ceramic. The following are the most common examples [10]:





These reactions occur at room temperature. By controlling the rate of reaction, ceramics can be formed. With trivalent oxides, similar ceramics can be formed at a slightly elevated temperature. A good example is berlinite (AlPO_4), which is formed by the reaction between alumina and phosphoric acid [10]:



2.1.2 Geopolymerization

Figure 2.1 presents a highly simplified reaction mechanism for geopolymerization. The reaction mechanism shown in Figure 2.1 outlines the key processes occurring in the transformation of a solid aluminosilicate source into a synthetic alkali aluminosilicate [11].

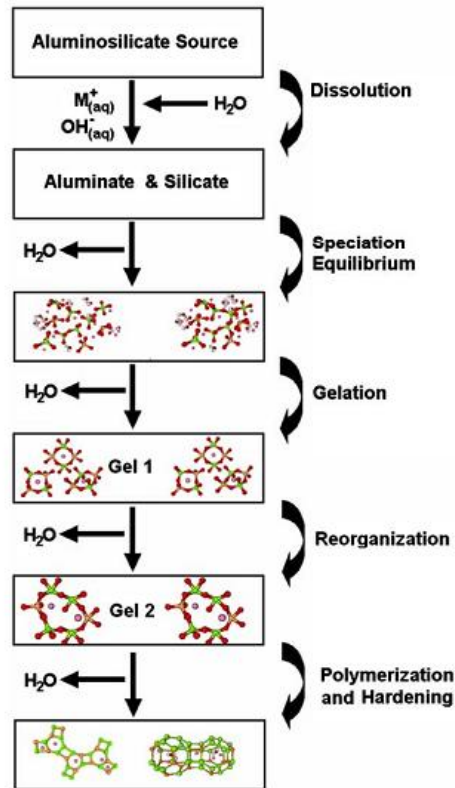


Figure 2.1: Conceptual model for geopolymerization [11].

Though presented linearly, these processes are largely coupled and occur concurrently. Dissolution of the solid aluminosilicate source by alkaline hydrolysis (consuming water) produces aluminate and silicate species. It is important to note that the dissolution of solid particles at the surface resulting in the liberation of aluminate and silicate (most likely in monomeric form) into solution has always been assumed to be the mechanism responsible for conversion of the solid particles during geopolymerization. Once in solution the species released by dissolution are incorporated into the aqueous phase, which may already contain silicate present in the activating solution. A complex mixture of silicate, aluminate and aluminosilicate species is thereby formed [11].

Dissolution of amorphous aluminosilicates is rapid at high pH, and this quickly creates a supersaturated aluminosilicate solution. In concentrated solutions this results in the formation of a gel, as the oligomers in the aqueous phase form large networks by condensation. This process releases the water that was nominally consumed during dissolution [11].

2.1.3 Cement Properties

Thickening Time

The thickening time is the length of time cement slurry will remain in a fluid state under simulated downhole condition without any shutdown periods [2]. Specific thickening time recommendations depend largely on the type of job, the well condition, and the volume of cement being pumped [2].

Temperature and pressure each influence the set of cement, whereas depths dictates the placement time (the greater the depth, the more time required). Pressure alone accelerates the setting of cement more in deep wells than was previously thought. This accelerating effect was not recognized until the development of super pressure/temperature testing apparatus for cementing wells to depths of 40,000 ft and at static temperature of 700°F [2].

Fluid Loss

In order to sustain a constant water/solids ratio the rate at which water is lost from a slurry under pressure must be controlled. Controlled water loss is preferred as slurries stay uniform and maintain constant properties during the time of their exposure to the formation [12].

Slurries become more viscous as they lose water. As a result, additional pump pressure is needed to move the slurry. This pressure can increase to the point that the formation could fracture, resulting in the possibility of lost circulation [12].

Bridging the annulus with dehydrated cement is considerably reduced with the use of an effective fluid loss control additive. If slurry is pumped past a permeable zone that is not protected by an adequate mud cake, flash setting may occur. If water is lost to such a zone the slurry may become a solid unpumpable mass. This situation is termed as a “flash set” [12]. The result is an abandoned job with cement still in the pipe. The risk of this situation is probably the most important reason for the use of fluid-loss additives in primary cementing.

Compressive Strength

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

Downhole parameters such as temperature and pressure also give effect to the compressive strength as these two parameters involve vitally during hydration of cement. Besides, water content, admixes and stirring time also give effect to the compressive strength of the cement. The theory behind compressive strength starts during static condition when gel strength takes places very rapidly within cement slurry

[2]. Figure 2.4 shows the development of compressive strength with respect to time and Figure 2.5 shows effect of temperature on hydration thus compressive strength.

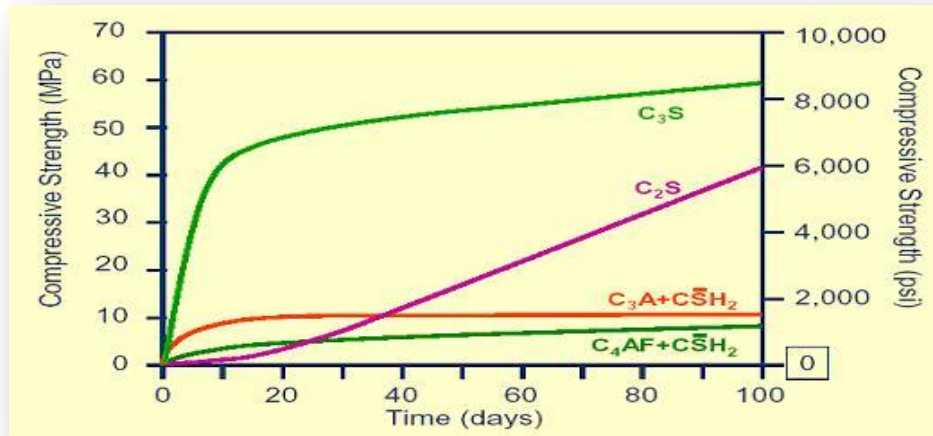


Figure 2.2: Compressive Strength Development Pure Cement [7].

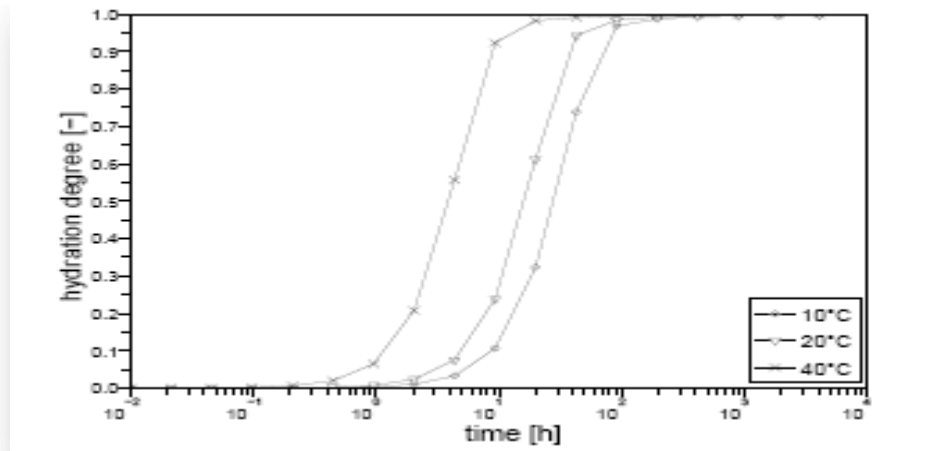


Figure 2.3: Influence of Temperature on Cement Hydration [9].

2.1.4 Cement Microstructure

The hydration products in cement paste do not exist in isolation, but are entangled with one another at the scale of microns and even nanometers. The possible exception to this is the high-density C-S-H phase that forms within the boundaries of the original cement grains [13]. Thus in describing the microstructure of cement paste the structure of the individual solid phases is not as relevant as the overall distribution of solid phases and porosity. With this in mind, a basic but useful description of cement paste microstructure is comprised of just three phases [13].

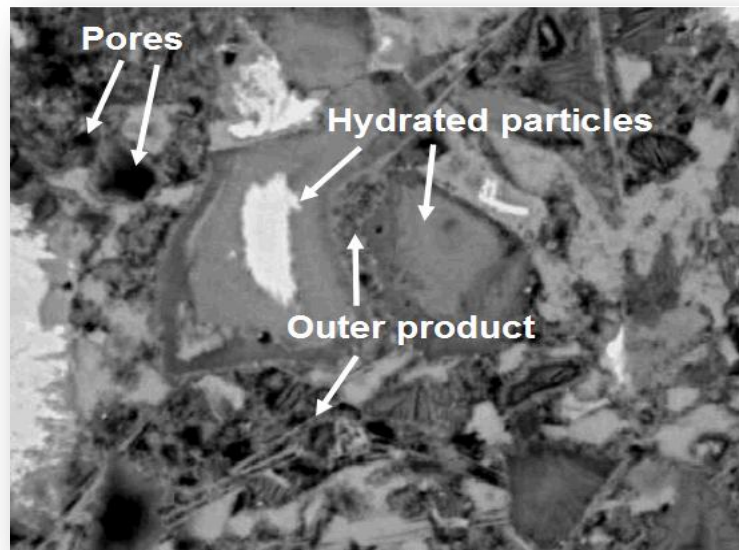


Figure 2.4: Main microstructure features [13].

Hydrated cement particles

These consist of high-density C-S-H and in some cases an interior core of unhydrated cement. These behave as individual solid particles within a continuous matrix analogous to the aggregate particles in concrete. These features are sometimes called "phenograins", which simply refers to the fact that they are distinctly visible in a microscope [13].

Outer hydration product

This is the continuous phase that grows within the capillary pore space and binds the cement together. Following the analogy used above, it plays a role similar to cement paste in concrete. It consists of solid C-S-H gel, gel pores, calcium hydroxide, and calcium sulfoaluminate phases. This "phase" appears as various shades of speckled grey in an optical or electron microscope, and is sometimes referred to as "groundmass." By far the most important individual phase is, of course, the low density C-S-H gel (and its gel pores), because it's high surface area gives this phase its strength [13].

Large pores

These consist of true capillary pores, entrapped air voids, and the entrained air system. These features appear as discrete black voids in a microscope. The large pore system can be continuous or discontinuous, depending on the degree of hydration and starting w/c, but this cannot be determined from microscopy [13].

2.1.5 Type of Cement

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

Table 2.2: The Difference Classes of API Cement for Use at Downhole Condition [2].

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

2.1.6 Additive in Cement Slurry

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

Cement Accelerators

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

Table 2.3: Common Accelerator in Cement Slurries [2]

Accelerator	Amount Used (wt% of Cement)
Calcium Chloride	2 to 4
Sodium Chloride	3 to 10
Gypsum-Hemihydrate form	20 to 100
Sodium Silicate	1 to 7.5
Cement Dispersant	0.5 to 1.0
Seawater (as mixing water)	-

Lightweight Additives

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

Table 2.4: Among Common Lightweight Additive in Cement Slurries [2]

Lightweight Cement Additives	Amount Used
Bentonite	2 to 16 wt% of Cement
Natural Hydrocarbon	
- Gilsonite	1 to 50 lbm/sack of cement
- Coal	5 to 50 lbm/sack of cement
Expanded Perlite	5 to 20 lbm/sack of cement
Nitrogen	0 to 70 wt% of Cement

Heavyweight Additives

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

Table 2.5: Among Common Heavyweight Additive in Cement Slurries [2]

Heavyweight Cement Additives	Amount Used (wt% of Cement)
Hematite	4 to 104
Ilmenite	5 to 100
Barite	10 to 108
Sand	5 to 25

Cement Retarder

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

Table 2.6: Among Common Retarder Additive in Cement Slurries [2]

Retarder	Amount Used (wt% of Cement)
Lignin retarder	0.1 to 1.0
Calcium lingo sulfonate, organic acid	0.1 to 2.5
Carboxy methyl hydroxyethyl cellulose	0.1 to 1.5
Saturated Salt Water	14 to 16 lbm/ sack of cement

Additives for Controlling Lost Circulation

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

Table 2.7: Among Common Lost Circulation Control Additive in Cement Slurries [2]

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

Filtration Control Agent

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

Table 2.8: Among Common Filtration Control Agent in Cement Slurries [2]

Filtration Control Agent	Amount Used (wt% of Cement)
Cellulose	0.5 to 1.5
Dispersant	0.5 to 1.25
Carboxy methyl hydroxyethyl cellulose	0.3 to 1.0
Latex additives	1.0 gal/ sack
Nylon	0.125 to 0.25 lbm/ sack

Friction Reducer

Friction reducer agents are added into cement slurries to improve the cement slurries flow properties.

Table 2.9: Among Common Friction Reducer Agent in Cement Slurries [2]

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

Special additives

Table 2.10: Among Common Special additives Agent in Cement Slurries [2]

Type	Amount Used (wt% of Cement)
Silica Flour	0.2 to 0.4
Dyes	0.1

2.1 Literature Review

Recent study [3] shows that the use of geopolymer in cementing reduces the emission of CO₂ and increased the mechanical properties of cement system. The new technology is based on a geopolymeric cement system incorporating amorphous aluminosilicate materials. A key attribute of this geopolymer system is its robustness and versatility which enables the product to be engineered from arrange of cement/aluminosilicate/fly ash component ratios so that it delivers specific properties for a given application at lowered cost.

Applications of particular interest at present include low or high density cement systems with enhanced mechanical properties and good chemical resistance for oil and gas well cementing. The new cement systems offer superior properties especially for low density slurries from 1200 to 1500 kg/m³. Therefore, they are regarded as replacements for traditional lightweight cements containing silica fume [3].

From the study by Amir H. Mahmoudkhani, SPE, Diana N.T. Huynh, Chuck Sylvestre, and Jason Schneider, Sanjel Corporation [3], the new geopolymer-cement system offers:

- Variable densities from 1200 to 1900 kg/m³
- Thickening times from several minutes to several hours.
- Superior early and late strength development.
- Fast gel strength development.
- Controlled fluid loss.
- Enhanced flexibility and elasticity.
- Zonal isolation through strong bonding to formation and casing.
- Ease of operation and handling.
- Compatibility with most common cements admixtures and additives.
- Significantly reduced CO₂ and water footprints.
- Cost savings.

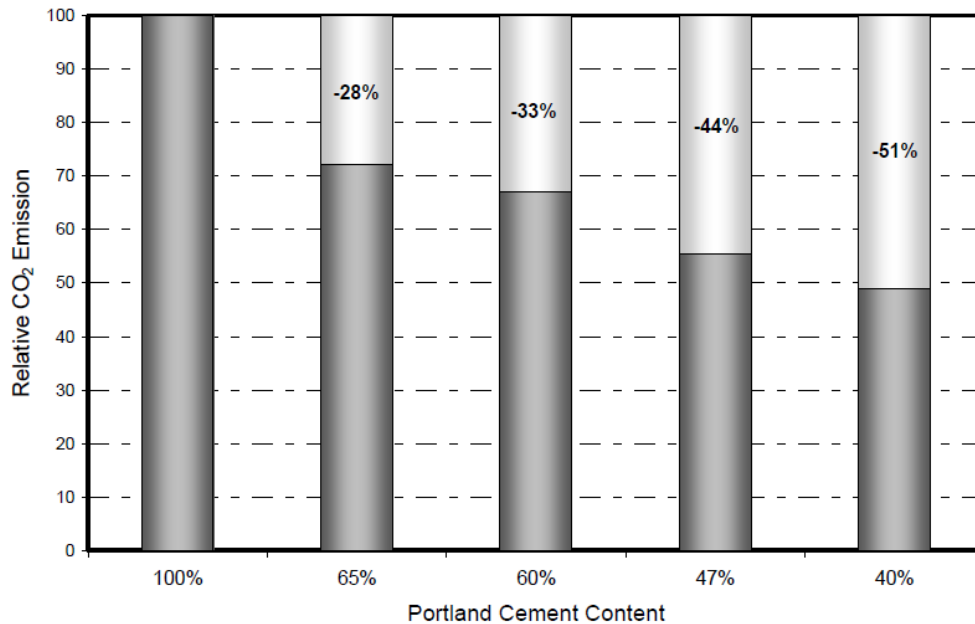


Figure 2.5: Resulted CO₂ emission [3].

Geopolymerization and Geopolymers

Geopolymerization is a general term used to describe all the chemical processes that are involved in reacting aluminosilicates with aqueous alkaline solutions to produce a new class of inorganic binders called geopolymers [3]. The geopolymeric reaction occurs as a result of reacting aluminosilicates with alkali and soluble alkali polysilicates. This reaction results in the formation of SiO₄ and AlO₄ tetrahedral linked by shared oxygen atoms [3].

A mild exothermic reaction in the alkali activated mixture is accompanied by hardening and polycondensation. Thus, a geopolymer can be described as a low calcium, alkali activated aluminosilicate cement. The structure is comprised of predominantly Si-O-Al and Si-O-Si bonds arranged in a solid X-ray amorphous aluminosilicate network. After long periods of curing at given temperature and pressure, the amorphous solid phase may transform into semi-crystalline phases [3].

Thickening Time and Static Gel Strength

The term transition time has been used to refer to the dynamic set profile of cement slurries under downhole conditions. Once slurry goes static, immediately after placement, it will start developing static gel strength (SGS) that will continue to increase until the cement is fully set. Slurries that provided a short transition time are those demonstrating a “right angle set” on a thickening time chart [3].

Transition time is the period during which the slurry changes from a true hydraulic fluid to a highly viscous mass showing some solid characteristics. By definition a “right angle set” is one in which the viscosity of the slurry remains relatively low through the majority of the test and then rapidly sets in a 20 to 45 minute time frame to more than 70 Bearden units of consistency (Bc) [3].

Right angle set together with static gel strength are important characteristics of cement slurries designed for controlling gas migration that may occur during cementing. In this regard, geopolymer cement blends are showing highly advantageous properties by having a fast set time [3].

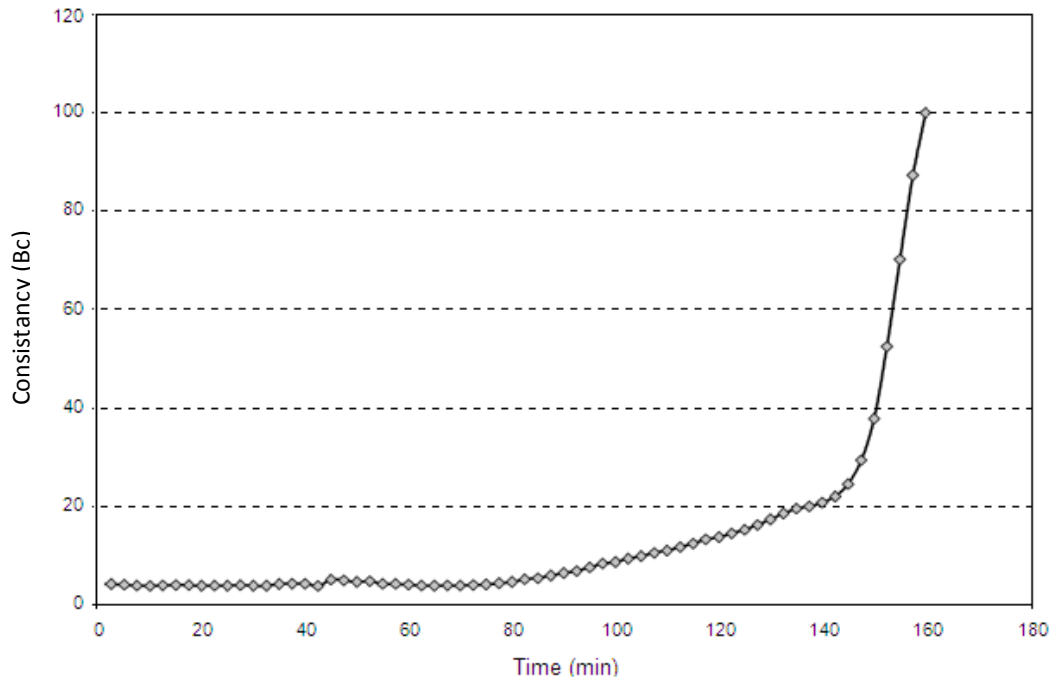


Figure 2.6: Thickening time profile for a geopolymer cement blend [3].

Table 2.11 : Gel strength of geopolymer cement slurries [3].

Additives	Density(kg/m ³)	BHST (°C)	Time to 100 lb/100ft ² (hh:mm)	Time to 500 lb/100ft ² (hh:mm)	Time to 1200 lb/100ft ² (hh:mm)	Transition Time(min)
None	1400	50	00:24	00:34	00:42	10
0.35%A1	1400	50	00:50	01:00	01:10	10
0.35%R1	1400	50	01:46	01:56	02:04	10
0.35%A1+0.35% R1	1400	50	03:06	03:18	03:28	12

A1=Free water control additive, R1= cement retarding additives

Compressive Strength

From the experimental result by Amir H. Mahmoudkhani, SPE, Diana N.T. Huynh, Chuck Sylvestre, and Jason Schneider, Sanjel Corporation [3], conventional lightweight neat cement blends show very low compressive strengths over the period of 48 hours, while geopolymer-cement blends perform significantly better.

During the investigation [3], the geopolymer cement shows superior early and late compressive strength development. The trend in compressive strength development compared to conventional neat cement is shown in Figure 2.10.

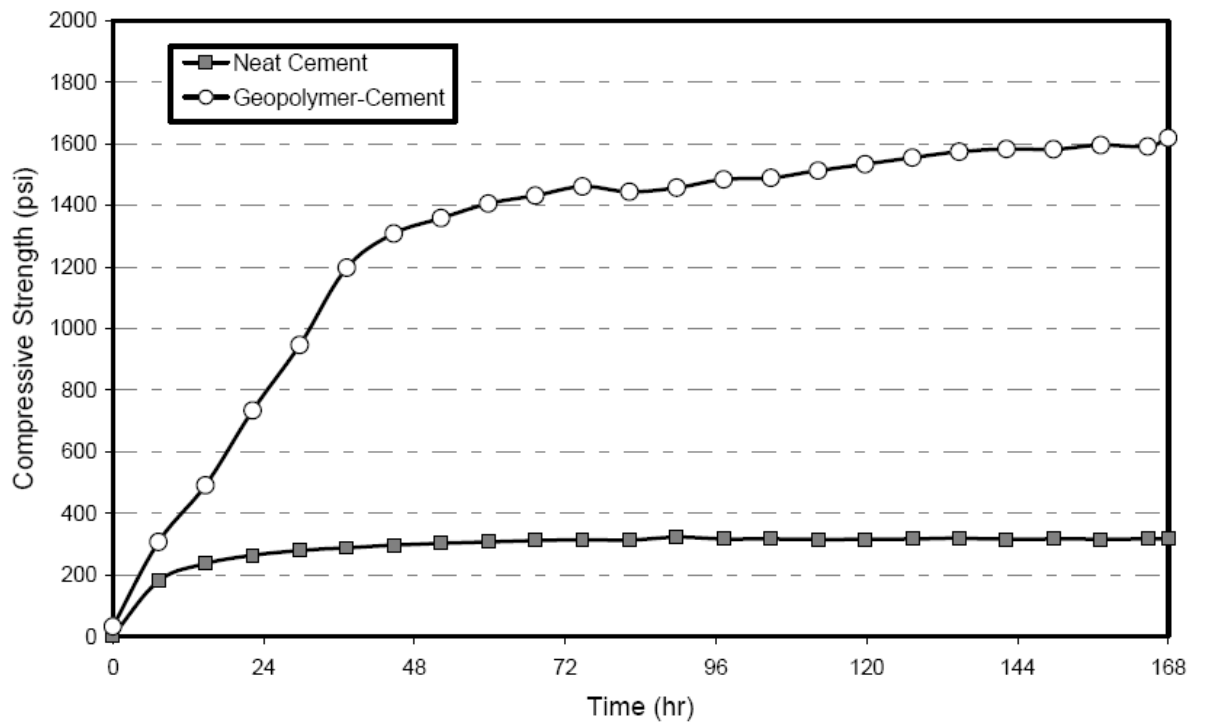


Figure 2.7: Compressive Strength Development [3].

CHAPTER 3

METHODOLOGY

3.1 Project Planning

3.1.1 Research Methodology

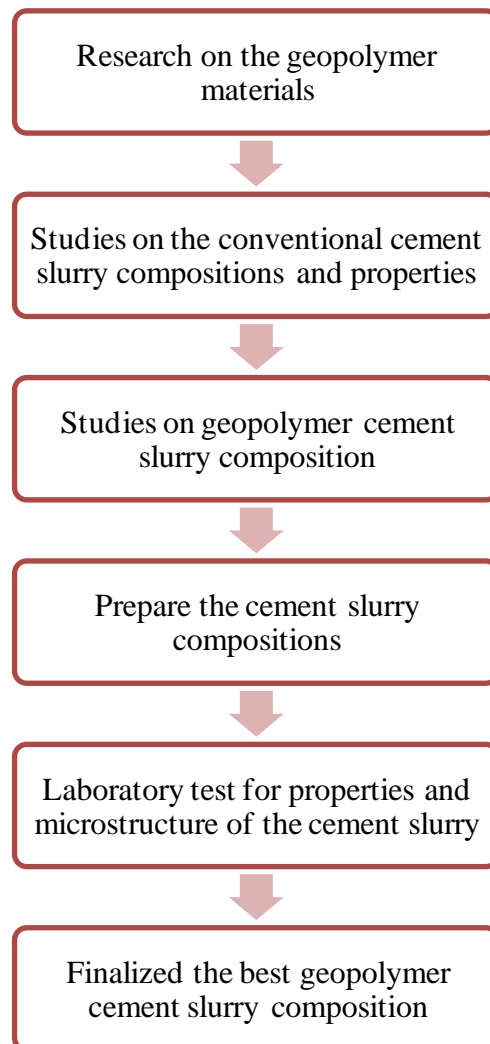


Figure 3.1: Flow Chart of the Research.

3.1.2 Gantt Chart and Key Milestone

Table 3.1: Gantt Chart and Key Milestone through the Final Year Project

Year	11									12
Activities	FYP 1					FYP 2				
	M	J	J	A	S	O	N	D	J	
Project planning and literature review										
Studies on geopolymers material										
Research on conventional cement slurry										
Studies on the factors affecting thickening time, fluid loss and compressive strength.										
Studies on designing geo-polymer cement composition										
Designing geopolymers cement slurry composition										
Measurement of properties of geopolymers cement slurry										
Comparison study with conventional cement slurry										
Research documentation										

Year	11									12
Milestone	FYP 1					FYP 2				
	M	J	J	A	S	O	N	D	J	
Completion of geopolymers material preparation										
Completion of conventional cement slurry composition and formulation										
Completion of measuring conventional cement slurry properties										
Completion of designing geopolymers cement slurries										
Completion of measuring geopolymers cement slurries properties										
Completion of geopolymers cement with improved properties										
Project completion										

3.1.3 Project Activities

Table 3.2: Project Activities through the Final Year Project

Activities	From Date	To Date
Studies on geopolymer material	1/07/2011	1/08/2011
Studies on conventional cement slurry composition	1/07/2011	1/08/2011
Studies on conventional cement slurry properties	1/07/2011	1/09/2011
Studies on geopolymer cement slurry composition	1/08/2011	1/09/2011
Measuring conventional cement slurry properties	1/09/2011	1/10/2011
Designing geopolymer cement slurry	1/09/2011	1/11/2011
Measurement of geopolymer cement slurry properties	1/10/2011	1/12/2011
Comparison study with conventional cement design	1/11/2011	1/012/2011
Research documentation	1/12/2011	1/01/2012

3.2 Experiment

In this research, five different cement slurries composition were prepared and tested with accordance to API RP-10B codes and standard. The cement samples are cured at temperature 200°F and pressure 3000 psi. The samples were prepared for each tested properties. Figure 3.2 shows flow chart of the experiment.

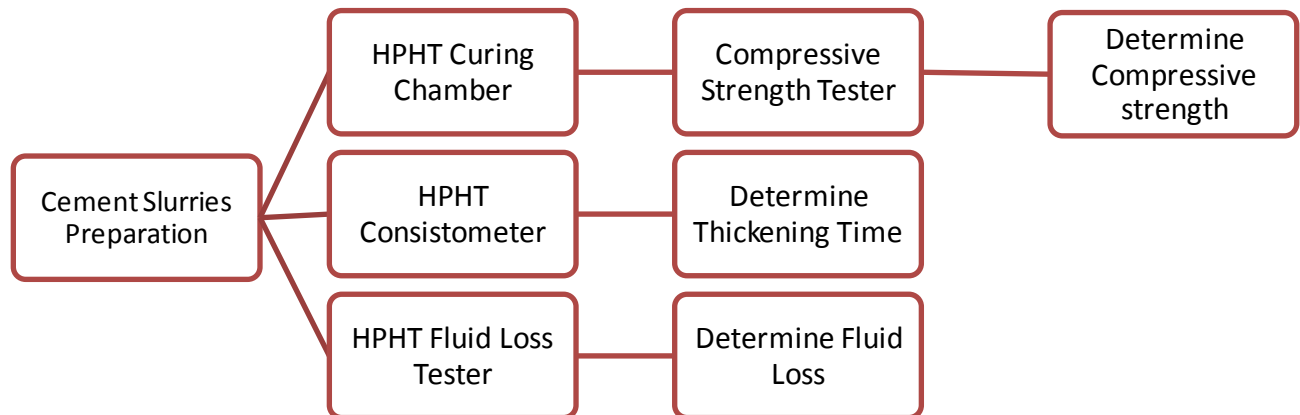


Figure 3.2: Flow Chart of Experiment.

3.2.1 Slurry Preparation

All of the cement slurries compositions were prepared at standard weight density. The amount of additives for every composition was made constant, added retarder and fluid loss additives were 1% and 8% by weight. Table 3.3 below shows slurries compositions and Figure 3.3 shows the preparation illustration.

Table 3.3: Cement Slurries Compositions.

Cement Slurry Samples	Compositions
Benchmark	Class G Cement + H ₂ O + Retarder + Fluid Loss Additive
Geo A	25% Fly Ash + 75% Class G Cement + Sodium Silicate + amorphous NaOH + Retarder + Fluid Loss Additive
Geo B	50% Fly Ash + 50% Class G Cement + Sodium Silicate + amorphous NaOH + Retarder + Fluid Loss Additive
Geo C	75% Fly Ash + 25% Class G Cement + Sodium Silicate + amorphous NaOH + Retarder + Fluid Loss Additive
Geo D	100% Fly Ash + Sodium Silicate + amorphous NaOH + Retarder + Fluid Loss Additive

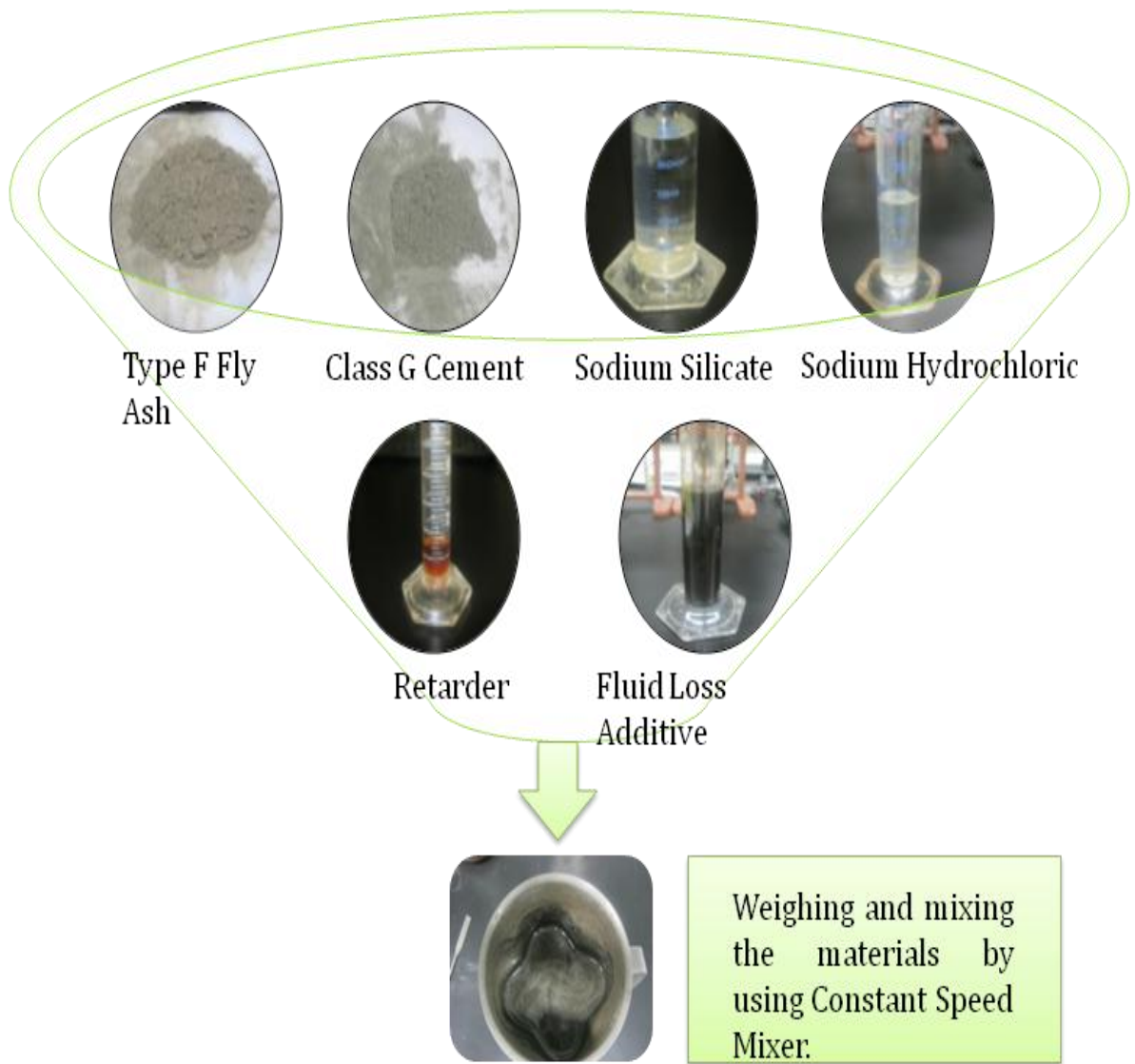


Figure 3.3: Slurry Preparation Illustration.

3.2.2 Properties Testing Preparation

Figure 3.4 below shows the preparation and flow for tested properties.

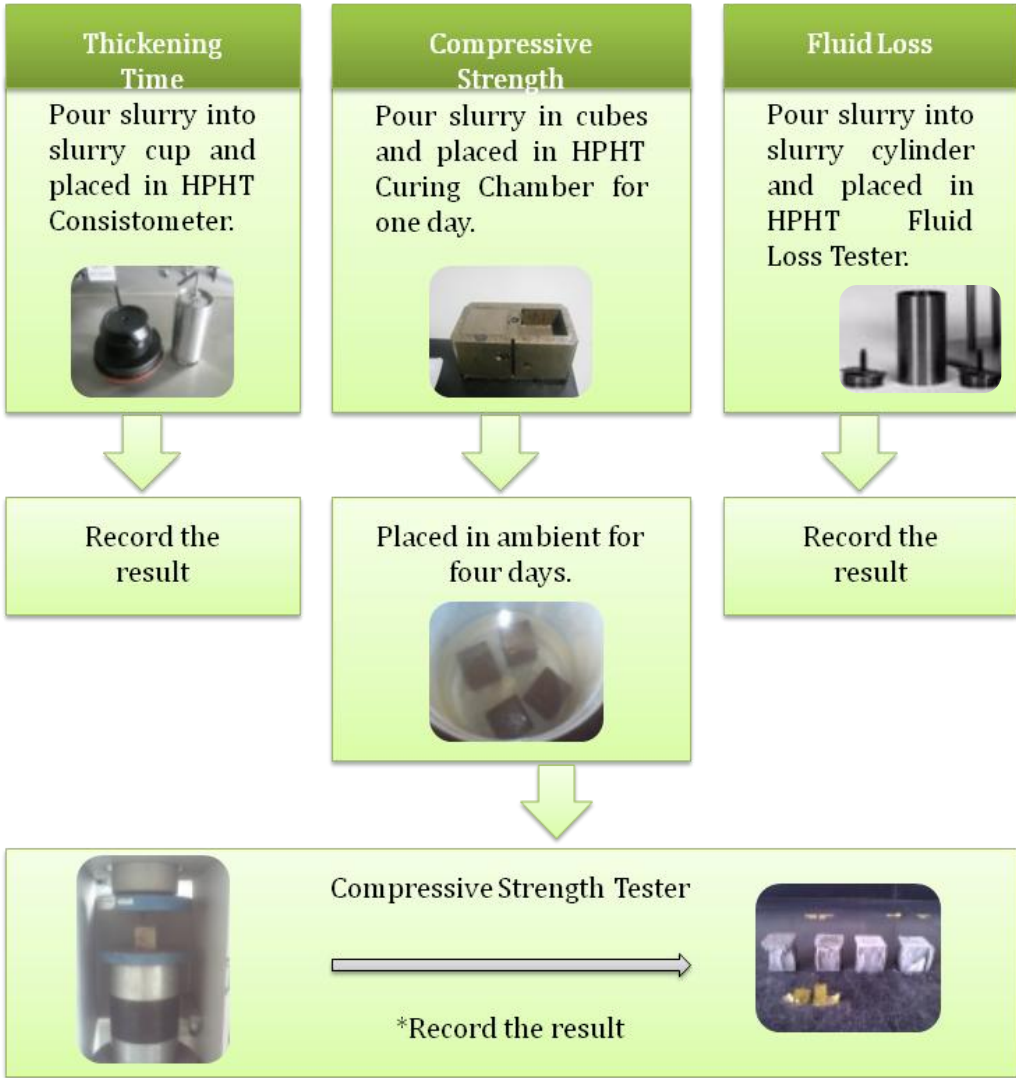


Figure 3.4: Flow of Testing Properties

3.3 Tools and Equipment

In this project, reference books and research paper are the essential source of data. Most of the books and research paper are available at the university's library. Required materials are below.

Table 3.4: Required Materials for Cement Slurry Design.

No	Material
1	API Class G Cement
2	Retarder additive
3	Fluid loss additive
4	Type F Fly Ash
5	Sodium Silicate
6	NaOH

Figure Below shows available equipments in Cement Laboratory under Geoscience and Petroleum Engineering Department.



Figure 3.5: Available equipments in Geoscience Laboratory.

CHAPTER 4

RESULTS AND DISCUSSION

This chapter will discuss the obtained results that were compared with the benchmark. The test results were presented and discussed further in this chapter.

4.1 Compressive Strength

The obtained results are shown in Table 4.1 below.

Table 4.1: Compressive Strength Results

Cement Slurry Samples	Compressive Strength (MPa)				Average Compressive Strength (MPa)
	A	B	C	D	
Benchmark	45.02	42.73	43.15	42.79	43.42
Geo A	51.03	53.30	55.43	50.11	52.47
Geo B	57.44	61.25	60.19	58.75	59.41
Geo C	63.22	64.79	64.65	61.78	63.61
Geo D	70.15	71.09	69.77	73.13	71.04

From the results, with the increasing addition of fly ash percentage in the compositions, the results yielded higher value of compressive strength. It is certain that the addition of fly ash in cement slurry composition do enhance the compressive strength of cement.

The reaction between fly ash, sodium silicates, and aqueous NaOH creates a polymer called geopolymer. The bonding of geopolymer enhanced the compressive strength of

the cement. It is observed that, percentage of geopolymer used is proportional to the increasing results and the use of 100% geopolymer gives the highest result. Thus, Geo D is the best composition for geopolymer cement for high compressive strength value.

In order to show clear comparison between samples, the results were presented in bar chart in Figure 4.1 below and Figure 4.2 shows the improvement in percentage.

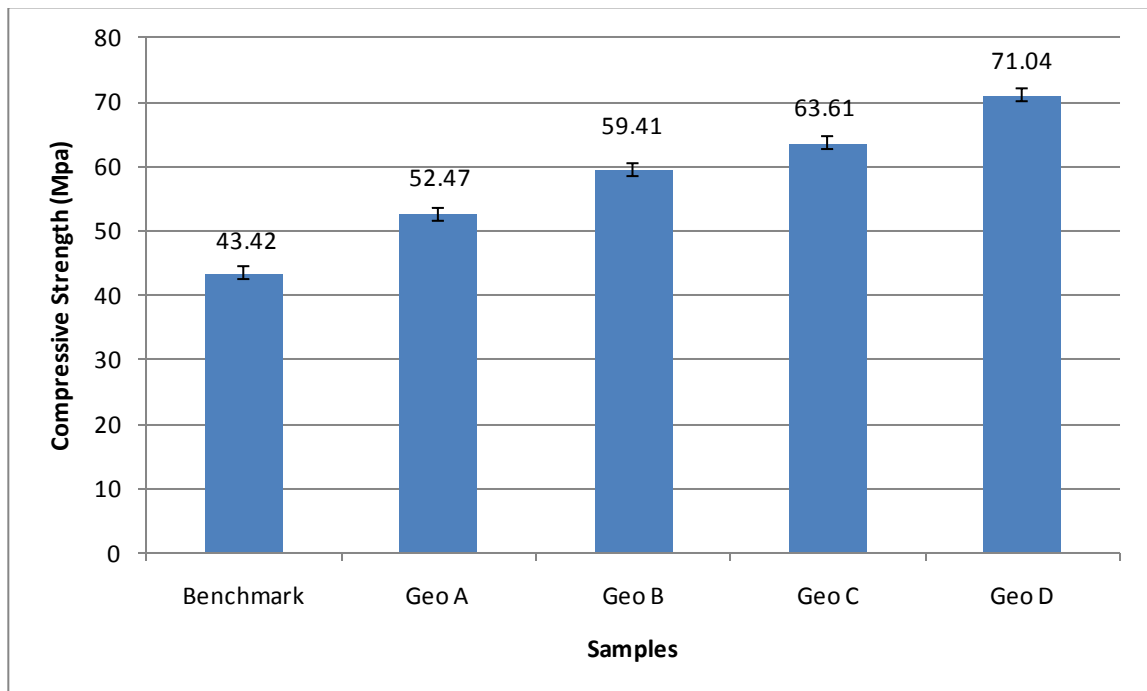


Figure 4.1: Compressive Strength Comparison

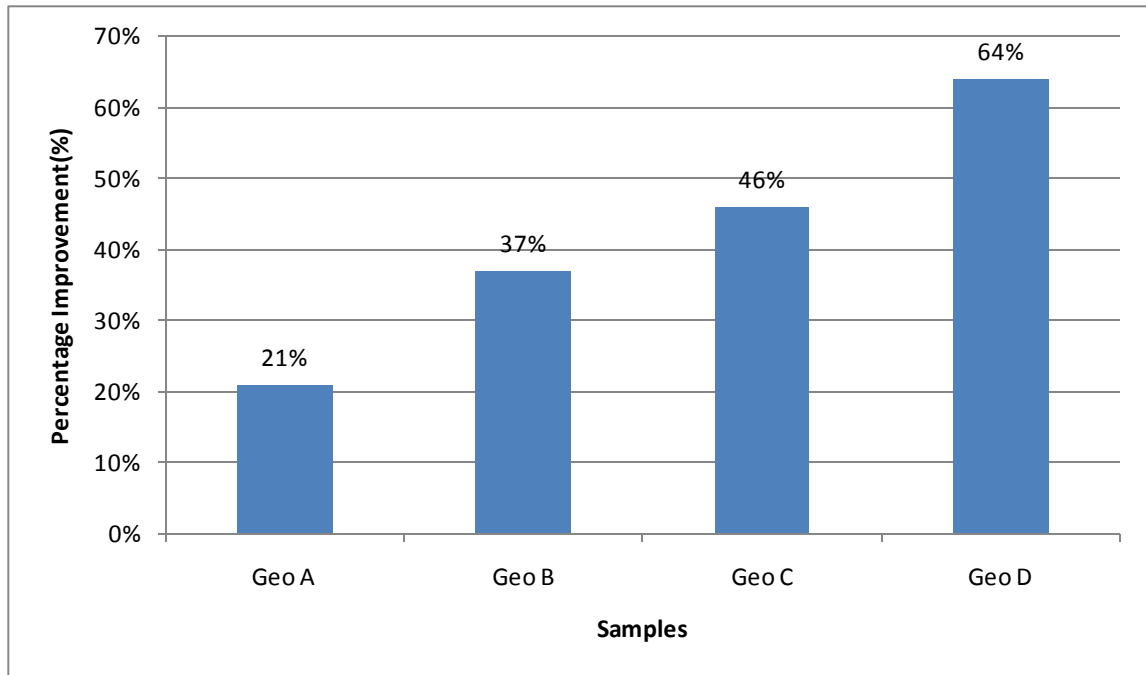


Figure 4.2: Compressive Strength Improvement

4.2 Thickening Time & Fluid Loss

Table 4.2: Fluid Loss and Thickening Time

Samples	Fluid Loss	Thickening Time (hour : min)	
		No Retarder	Retarder
Benchmark	94 cc/30 min	1:54	3:57
Geo A	81 cc/30 min	1:46	3:44
Geo B	74 cc/30 min	1:33	3:39
Geo C	62 cc/30 min	1:26	3:22
Geo D	55 cc/30 min	0:53	3:12

For the cementing activity, the adequate thickening time for cement slurry is between 3 to 4 hours due to the pumping of cement to the down hole. The thickening time is

important for the efficiency of pumping activity. All of the compositions were tested with no use of retarder additive and then tested with 1% by weight of retarder additive to show the differences. The purpose is to identify whether geopolymer cement's thickening time can be controlled with additive.

From the yielded results, it shows that the thickening time of the geopolymers can be controlled by using the additive. By adding retarder additive, the thickening time of all compositions have been retarded to the range 3-4 hours which are needed in pumping activity. Thus, all of the compositions are applicable for oil well job.

For fluid loss properties, all of the compositions contained 8% by weight of fluid loss additive. Because of the principal on how fluid loss additive works which are forming films or micelles and improve particle-size distribution, it will work on any type of bonding of cement. Thus, it is applicable in geopolymer cement.

From the result, as the nature of fly ash particle is very small and fine, the size and volume of pores have been reduced and easily equally distributed, the value of fluid loss decreasing as the amount of geopolymer increased in the compositions. Geo D where 100% of its composition is geopolymer yielded the best result in the fluid loss test. Clearer comparison showed in the Figure 4.3 and percentages of improvement are showed in Figure 4.4.

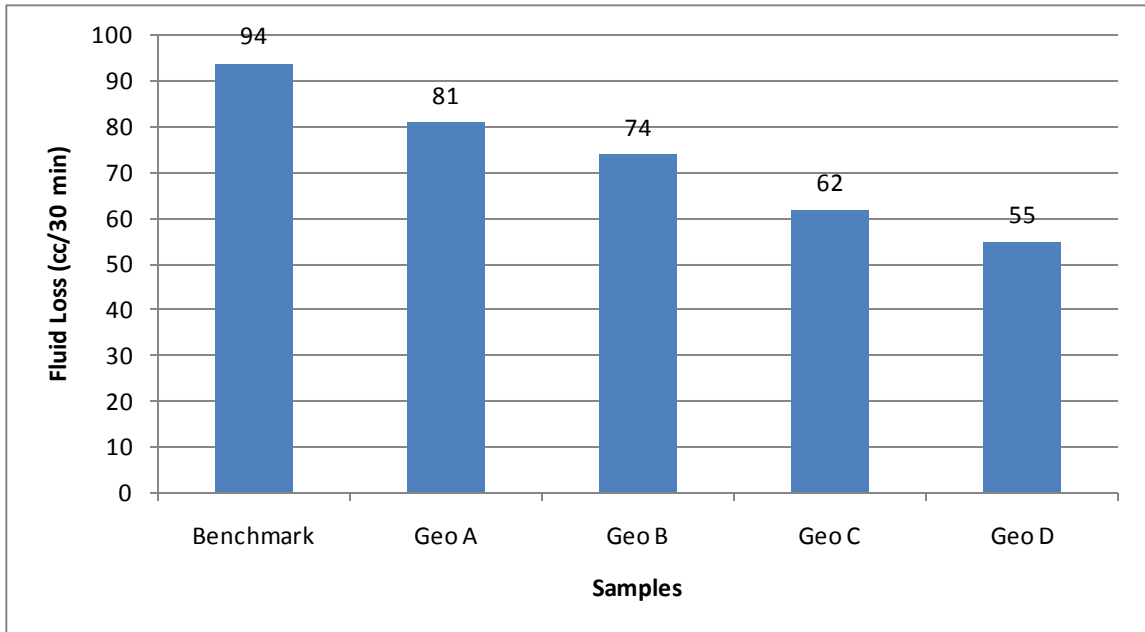


Figure 4.3: Fluid Loss Comparison

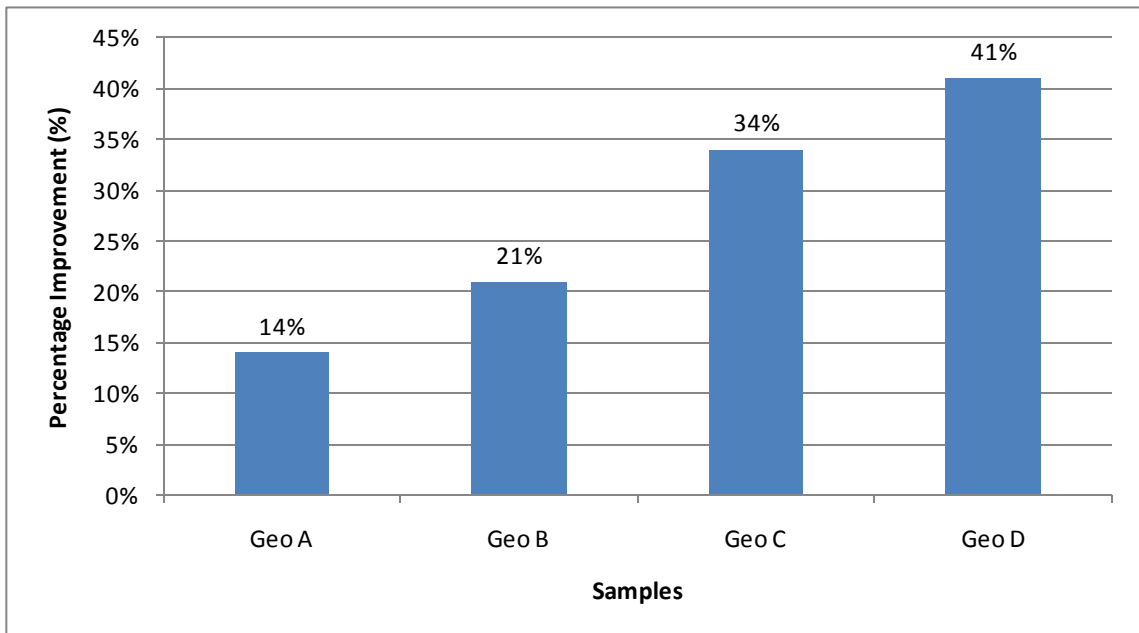


Figure 4.4: Fluid Loss Improvement

CHAPTER 5

CONCLUSION & RECOMMENDATION

From the obtained data, the following conclusions can be drawn:

- Geo D where 100% composition is geopolymer cement is the best slurry composition for geopolymer cement with improved properties. The objectives of the research achieved and also indicate that geopolymer cement can be used in oil well cementing.
- Geo D showed 64% and 41% improvement in compressive strength and fluid loss from the conventional cement.
- The bonding of cement slurry was proven to increase with the presence of geopolymer in the slurry compositions where with the increasing of geopolymer percentage in slurry composition proportionally to the increase of compressive strength value.
- The thickening time of geopolymer cement can be controlled by using additive. From the experiment, by using retarder the thickening time of geopolymer cement is longer.

Recommendation:

- This research on geopolymer cement should be continued because this technology has not yet matured and lots of findings can be found.
- For this research, the source of geopolymer is fly ash. It can be extended by implying new source of geopolymer cement..
- In addition, the research can be extended by testing the geopolymer in different oil well conditions.

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