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Formulation of Geopolymer Cement using Class F Fly Ash for Oil Well Cementing application

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Abstract

With the increasing awareness of global warming, there is a dire need to find a substitute for the conventional Ordinary Portland Cement (OPC) which produces huge amounts of carbon dioxide (CO_2) during its production. Class F fly ash based geopolymer cement has been identified as a potential replacement due to its abundant availability and to address waste management issues. The lack of research and a standard formulation for geopolymer cement has hindered the efforts to apply geopolymer cement for oil well cementing operations. Previous researches in the area of geopolymer cement has been focused mainly on its applications in the construction industry. Therefore, there is a strong need to develop a formulation of class F fly ash based geopolymer with varying parameters, ratios and compositions for oil well cementing applications. A total of 36 formulations were tested for density, rheology, fluid loss, thickening time and compressive strength according to API cement testing procedures by varying alkali concentration, fly ash to alkali binder ratio and sodium silicate to sodium hydroxide ratio. The results suggest that class F fly ash based geopolymer cement could be a better replacement for OPC cement in oil well cementing applications at high pressure and temperature conditions.

Keywords: fly ash, geopolymer cement, oilwell cement, thickening time, fluid loss, compressive strength

INTRODUCTION

The primary objective of oil well cementing is to prevent fluid migration from the formation and to anchor the casing onto the wellbore. Besides that, it provides zonal isolation and prevents movement of fluid between consecutive formations. The cementing process is pivotal in oil and gas industry as it is a high risk operation which requires proper planning and execution. Most oil well cementing applications are performed using different classes of OPC with varying additives according to its required application and wellbore conditions. It was found that approximately 0.66 to 0.82 kg of CO_2 is released to the atmosphere for the production of 1 kg of OPC and it contributes to 5-7% of the global anthropogenic CO_2 emissions [1]. In addition, the CO_2 emission is expected to increase and reach approximately 77Gt/ year by 2100 with an average atmospheric CO_2 concentration of approximately 750 parts per million by volume [2]. Due to the increasing awareness in addressing this issue, viable replacement for the conventional Portland cement is currently being reviewed and studied in detail. Besides that, studies have shown that there are several problems associated with the use of Portland cement for drilling applications, such as degradation of well cement, susceptibility to chemical reactions, poor durability and leakage [2-4]. Therefore, there is a dire need to develop a sustainable cement technology which possesses superior properties compared to the OPC for oil well cementing. With a reduced carbon footprint, geopolymer cement has been found to possess superior edge over OPC for oilwell cementing applications [2, 5-7]. However, the optimum formulation which yields superlative performance of geopolymer cement has to be determined. This research focuses on developing a formulation for class F fly ash based geopolymer cement for the oilwell cementing applications.

FLY ASH BASED GEOPOLYMER CEMENT

Geopolymer cement is an inorganic binder which can be polymerized from materials which are rich in silica and alumina. It is synthesized under high alkaline condition from alumina silicate polymers and alkali silicate solutions which consist of amorphous and three dimensional structures through the geopolymerization of alumina silicate monomers in alkaline solution [6]. Upon the synthesis, geopolymers should ideally consist of alumina and silica tetrahedral interlinked in an alternating manner whereby oxygen atoms are shared among the alumina and silica atoms. The introduction of the alumina silicate polymers in the alkaline solution initiates the geopolymerization process and the slurry begins to harden quickly. The short settling and hardening time which enhances its mechanical properties is due to its tightly packed polycrystalline structure. The synthesis of geopolymer is greatly attributed to the ability of the aluminum ions to initiate chemical changes in the silica backbone [8]. For the synthesis of geopolymer cement. the source of alkaline chemicals are usually $Ca(OH)_2$, NaOH, Na_2SiO_3, the combination of NaOH and Na_2SiO_3, the combination of KOH and NaOH, K_2SiO_3 and its combination, and $NaCO_3$. Different combinations of alkaline solutions would yield in different strengths and properties associated with the geopolymer cement. In general, materials which is made up of high compositions of Alumina and Silica in its amorphous form are suitable to be used as geopolymer cement. This underlines the benefit of using industrial by-product materials which are rich in Alumina and Silica such as fly ash, slag and rice husk as binders for the formulation of geopolymer cement. This study focuses on the low calcium fly-ash based geopolymer cement (class F) which is a byproduct from the combustion of coal in coal power plants. The current annual fly ash production is estimated to be 500 million tonnes and only 16 % of the total fly ash produced are currently being utilized[9]. With an annual increase of coal consumptions for power generation, the over production and underutilization of fly ash possess a serious threat to the environment as it is merely disposed in landfills.

FORMULATION OF CLASS F FLY ASH BASED GEOPOLYMER CEMENT

The lack of experimental work performed in the area of class F fly ash based geopolymer cement prompted this research idea to investigate the optimum formulation to cure the fly ash based geopolymer cement to produce superior properties for oil well cementing applications. Alkaline activators are required in the preparation of fly ash based geopolymer cement. The activation of fly ash would depend on the type of activation solution used. The activation solution which contains soluble silicates in them such as sodium or potassium silicate, would result in quicker mechanical strength development due to higher reaction rates compared to the usage of hydroxides alone as the activator solution [10]. However, there are no clear experimental results which distinguish the better option between NaOH and KOH on their effect on the reaction rates of Fly Ash [10]. In most cases, researches preferred to use NaOH compared to KOH since it is cheaper and widely available. The most commonly used alkaline solution is the combination of NaOH solution and Na2SiO3 solution. In most experiments conducted, alkali activating solution such as NaOH and KOH are added to Na2SiO3 which serves as a stimulating tool to improve the alkalinity of the solution, hence resulting in higher compressive strengths [11, 12]. It was found that the ratio of 1:2.5 of NaOH to Na2SiO3 resulted in the highest compressive strength for fly ash based geopolymer compared to the ratios of 1:1.75, 1:2, 1:2.25, and 1:3 [13].

Moreover, the concentration of NaOH also plays a pivotal role in the synthesis of fly ash based geopolymer cement. It was found that the compressive strength increases when the concentration of the NaOH in the solution is increased irrespective of liquid/fly ash ratio [14].However, in a study conducted by Joe, M et al [15] the optimum NaOH molarity 12M gave the highest compressive strength compared to NaOH molarity of 8M, 10M, 14M and 16M. Based on their experimental analysis, it was found that the fly ash geopolymer cement specimen with 12M NaOH gave 1.25 times higher compressive strength compared to other molarities [15].

In addition, the amount of water used in the formulation of the geopolymer cement affects its properties upon curing. Jaarsveld et al. [16] studied the effect of water content on the compressive strength for geopolymer cement and found that the optimum water/fly ash ratio was 0.43 for both alkali activating solution of NaOH and KOH. However, in another study conducted by Ghosh,K et al.[17] , it was found that the optimum water/fly ash ratio was 0.3 and increasing the water content beyond the optimum value resulted in a lower compressive strength value. In both cases the curing regime was different which resulted in different optimum values. However, in this study, the optimum value of water/fly ash ratio has to be determined mimicking wellbore conditions. For this study, the quantity of water is defined as the total sum of water contained in the NaOH, Na2SiO3 activator and also the amount of added water. Moreover, the effect of adding dispersants, which is used to improve the rheological properties of the cement slurry was also examined in this study.

The temperature at which the geopolymer cement is cured plays a pivotal role in achieving the final compressive strength. Many authors have reported that the rate of fly ash geopolymerization reaction increases as the curing temperature increases until the optimum curing temperature is reached [2, 7, 18-20]. Besides that, the synthesis of geopolymerization process would require a temperature ranging from 20oC to 80oC which was one of the basis whereby many experiments were conducted in that temperature range. However, studies have shown that the fly ash geopolymerization reaction at ambient temperatures is extremely slow and results in a very low compressive strength [2, 8, 14]. Therefore, the temperature profile of the well has to be studied accordingly as it would not be practical to provide heat curing for the entire length of the wellbore in cases where the temperatures are below 23oC. According to the current industry practices, the most suitable curing pressure for the oil and gas wells is from 1000 to 3000psi. This pressure range is chosen because it is the pressure range encountered in normal reservoir conditions.

Apart from curing temperature and pressure, the curing time is an important factor for the development of compressive strength of fly ash based geopolymer cement. The curing duration is analogous to the thickening time whereby the thickening time of oil well cement is a function of mixing and pumping time, displacement time and plug release time. The experimental results carried out by most researchers shows that the curing time is dependent on curing temperature and similar trend was observed as in the curing temperature analysis whereby the compressive strength reduces after an optimum curing time [7, 10, 18, 20]. However, in another study conducted, it was found that curing time of 24 hours and 48 hours does not influence the compressive strength of the geopolymer cement rapidly which suggests that curing for 24 hours is adequate [13]. Therefore, curing for 24 hours is sufficient for oilwell cementing applications as longer curing time translates to higher costs and possibilities of lost circulations during drilling operations.

EXPERIMENTAL METHODOLOGY

The first part of the experiment consists of formulating the class F fly ash based geopolymer cement. The slurries were formulated by varying the NaOH molarity, fly ash to alkali ratio and NaOH to Na_2SiO_3 ratio. Thirty-six different compositions of slurries with different molarities of NaOH (10M, 12M and 14M) were tested. For each molarity, the slurry was formulated with different fly ash to alkali ratio (60:30, 45:45, and 30:30) and NaOH to Na_2SiO_3 ratio (2.5, 1, 0.5, and 0.25). In all cases, the amount of water added was kept constant at 10% of the overall weight. Table 1,2 and 3 illustrates the different compositions for respective molarities of NaOH.

The total volume of cement slurry for all the cases were kept constant at 600mL. The NaOH solution was prepared by diluting it with deionized water. Upon diluting the NaOH solution to the selected molarity, the Na2SiO3 solution was then added into the NaOH solution, followed by fly ash and 10 grams of dispersant. The mixture was mixed in the mechanical mixture at 1200RPM for 1 minute. Using a mud balance, the density of the cement slurry was tested. The density of the slurry should be in the range of 12.5ppg to 16ppg. Later, the slurry rheology was measured using a viscometer cup with heating elements to ensure that the readings were taken at 80ºC resembling reservoir conditions. The plastic viscosity of the cement slurry should be more than plastic viscosity of the drilling mud and the yield point should be more than 5.The slurries which passed the fluid rheology and density requirements were then tested for fluid loss using a HPHT fluid loss tester. The fluid loss tester was set at 125ºC and 1000 psi to simulate the rate of water loss in wellbore conditions. According to the industry requirement, the fluid loss should be in the range of 60 to 120 mL in 30 minutes. The final test performed was the compressive strength test. Cement samples were transferred into the curing molds of 50mmx50mmx50mm and cured at 80°C and 2000psi for 24 hours. The cured cement cubes were then tested for its compressive strength.

Table 1. Slurry composition with 10M NaOH.

Composition	1	2	3	4	5	6
Na2SiO3/NaOH		2.5			1	
Fly Ash : Alkali	60:30	45:45	30:60	60:30	45:45	30:60
Fly Ash (g)	360	270	180	360	270	180
Alkali (g)	180	270	360	180	270	360
NaOH (g)	51	77	103	90	135	180
Na2SiO3 (g)	129	193	257	90	135	180
Composition	7	8	9	10	11	12
Na2SiO3/NaOH		0.5			0.25	
Fly Ash : Alkali	60:30	45:45	30:60	60:30	45:45	30:60
Fly Ash (g)	360	270	180	360	270	180
Alkali (g)	180	270	360	180	270	360
NaOH (g)	120	180	240	144	216	288
Na2SiO3 (g)	60	90	120	36	54	72

Table 2. Slurry composition with 12M NaOH.

Composition	13	14	15	16	17	18
Na2SiO3/NaOH		2.5			1	
Fly Ash : Alkali	60:30	45:45	30:60	60:30	45:45	30:60
Fly Ash (g)	360	270	180	360	270	180
Alkali (g)	180	270	360	180	270	360
NaOH (g)	51	77	103	90	135	180
Na2SiO3 (g)	129	193	257	90	135	180
Composition	19	20	21	22	23	24
Na2SiO3/NaOH		0.5			0.25	
Fly Ash : Alkali	60:30	45:45	30:60	60:30	45:45	30:60
Fly Ash (g)	360	270	180	360	270	180
Alkali (g)	180	270	360	180	270	360
NaOH (g)	120	180	240	144	216	288
Na2SiO3 (g)	60	90	120	36	54	72

Table 3. Slurry composition with 14M NaOH.

Composition	25	26	27	28	29	30
Na2SiO3/NaOH	2.5			1		
Fly Ash : Alkali	60:30	45:45	30:60	60:30	45:45	30:60
Fly Ash (g)	360	270	180	360	270	180
Alkali (g)	180	270	360	180	270	360
NaOH (g)	51	77	103	90	135	180
Na2SiO3 (g)	129	193	257	90	135	180
Composition	31	32	33	34	35	36
Na2SiO3/NaOH	0.5			0.25		
Fly Ash : Alkali	60:30	45:45	30:60	60:30	45:45	30:60
Fly Ash (g)	360	270	180	360	270	180
Alkali (g)	180	270	360	180	270	360
NaOH (g)	120	180	240	144	216	288
Na2SiO3 (g)	60	90	120	36	54	72

RESULTS AND DISCUSSION

Slurry Density and Rheology Test

The cement slurries were prepared according to the formulation of geopolymer cement explained in the methodology. The results of the slurry density and rheology tests are tabulated in Table 4. For the compositions with 10M of NaOH (Compositions 1-12), the all formulations failed either in the slurry density or rheology test. Most of the formulations resulted in slurries which thickened very fast and became gelled quickly. On the other hand, for compositions with 12M of NaOH (Composition 13-24), the formulations with 60:30 ratios of fly ash to alkaline activator (Composition 13,16,19 and 22) passed the minimum requirement of the slurry density and rheology test. The other ratios of fly ash to alkaline activator resulted in lower density and failed slurry rheology due to low amount of fly ash which is the primary material for the formation of geopolymer chains. Lastly, compositions with 14M of NaOH (Compositions 25-36) also failed in the slurry density and rheology tests. Moreover, compositions 28 and 30-36 were completely gelled which prior to the first test itself. This can be attributed to the high molarity if NaOH which releases high amounts of heat during the binding process which results in a very quick fly ash geopolymerization process. Based on the slurry density and rheology test results, only compositions 13, 16, 19 and 22 passed the requirements and were tested for the fluid loss, thickening time and compressive strength.

Table 4. Slurry Density and Rheology Test Results for Compositions 1-36

Composition	1	2	3	4	5	6	7	8
Density (ppg)	15.2	14.2	13.6	15	12.5	13.5	Gelled	Gelled
PV (cP)	109.5	56.25	97.5	Gelled	28.5	21	Gelled	Gelled
YP (lb/ft2)	-0.5	1.75	-12.5	Gelled	1.5	2	Gelled	Gelled
Composition	9	10	11	12	13	14	15	16
Density (ppg)	Gelled	15	13.7	12.2	15.8	14.8	13.7	15.5
PV (cP)	Gelled	34.5	21	9	195	133.5	105	172.5
YP (lb/ft2)	Gelled	6	7	2.5	5	3.5	2	9.5
Composition	17	18	19	20	21	22	23	24

Composition	1	2	3	4	5	6	7	8
Density (ppg)	14.4	13.5	15.4	14.1	13.6	15.2	14.2	12.4
PV (cP)	49.5	24	127.5	46.5	31.5	88.5	28.5	15
YP (lb/ft2)	1.5	3	12.5	1.5	1.5	5.5	1.5	1
Composition	25	26	27	28	29	30	31	32
Density (ppg)	15.7	14.1	12.7	Gelled	13.5	Gelled	Gelled	Gelled
PV (cP)	204	99.75	54.75	Gelled	70.5	Gelled	Gelled	Gelled
YP (lb/ft2)	1	2.25	2.75	Gelled	1.5	Gelled	Gelled	Gelled
Composition	33	34	35	36				
Density (ppg)	Gelled	Gelled	Gelled	Gelled				
PV (cP)	Gelled	Gelled	Gelled	Gelled				
YP (lb/ft2)	Gelled	Gelled	Gelled	Gelled				

Table no.4 continued….

Fluid Loss Test

The fluid loss test was conducted on the formulation of fly ash based geopolymer cement which passed the slurry density and rheology test. The equipment was set at 260 ºF and 1000 psi to simulate the rate of water loss at downhole conditions

According to industry requirements, the fluid loss should be within 60ml to 120ml in 30 minutes' interval. 1 shows the amount of fluid loss (ml) in 30 minutes for composition 13, 16, 19 and 22. The test was conducted at 1000 psi and 260 °F to simulate downhole conditions. The results of the fluid loss tests are summarized in the Figure 1.

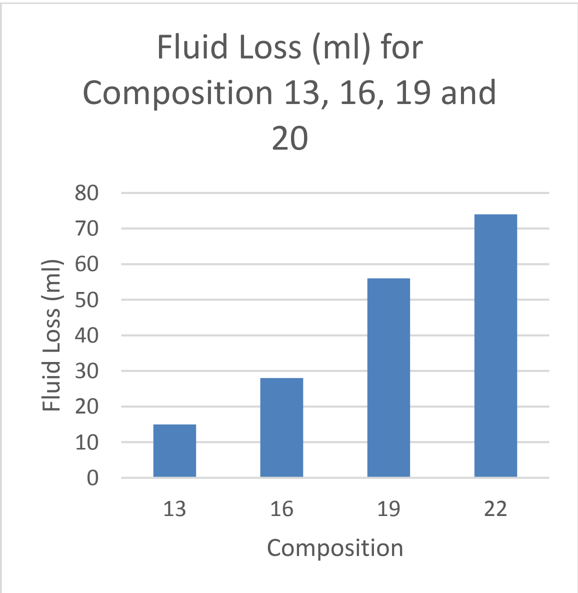

Figure 1. Amount of fluid loss in composition 13, 16, 19 and 22

From Figure 1, it can be concluded that composition 22 falls within the range of the industry requirement which allows a filtrate volume of 60 ml – 120 ml within the 30-minute interval. However, for tight gas well applications, compositions 13, 16 and 19 can be considered as the filtrate volume requirements are below 30 ml within a 30-minute interval. Next the thickening time test was conducted for all four compositions which passed the minimum requirement in the slurry and density test.

Thickening Time Test

The thickening time test was conducted using a HPHT Consistometer. The thickening time requirement for general oil well cementing applications is 40 Bc in 4 hours. Figure 2 shows the consistency achieved for composition 13, 16, 19 and 22 at a 4-hour interval.

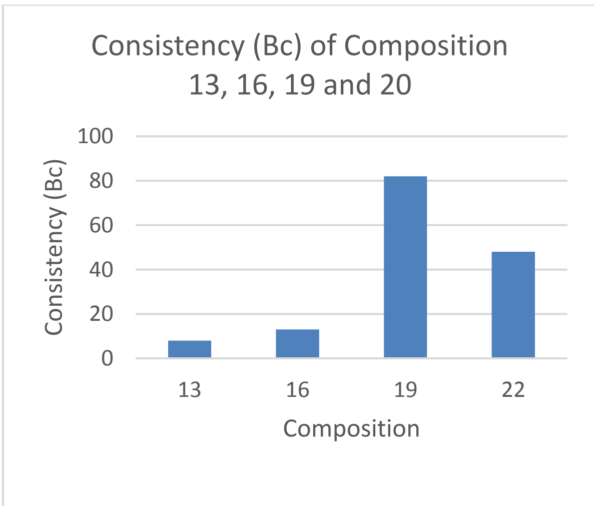

Figure 2.Consistently achieved for composition 13, 16, 19 and in 4 hours.

The result for thickening further shows that the optimum composition is composition 22 as it produces a consistency of 48 Bc in 4 hours. In most cases, a thickening time of 30-70 Bc is required to enable adequate pumping time for oil well cementing applications. Insufficient thickening time would result in the setting of cement at different depth from the designed depth.

Compressive Strength Test

The compressive strength test was conducted for composition 22 as it passed all the requirements set in the slurry rheology and density test, fluid loss test and thickening time test. The compressive strength test was performed using a compressive strength tester. The cement slurry of composition 22 was prepared and cured for 24 hours in the HPHT curing chamber prior to performing the compressive strength test. In addition, the curing conditions were varied at curing temperatures of 60 °C, 80 °C and 100°C and curing pressures of 1000psi, 2000psi and 3000psi to observe the effects of curing temperature and pressure on the compressive strength of the cement cubes. Figure 3 shows the compressive strength of composition 22 under different curing temperatures and pressures.

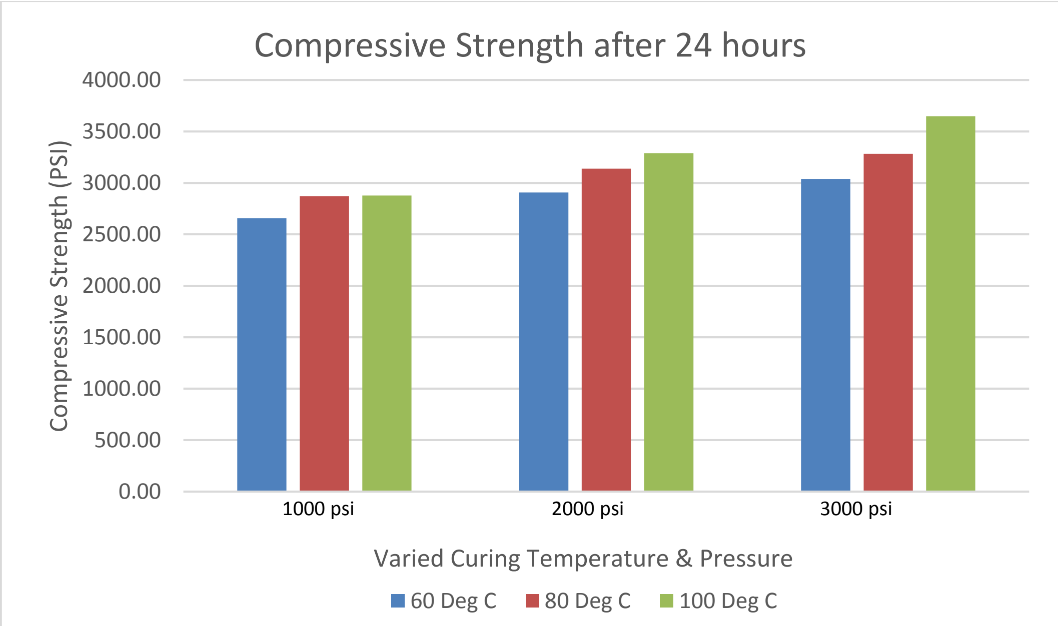

Figure 3. Compressive strength of Composition 22 at different curing temperatures and pressures.

Based on the results obtained, it is evident that class F geopolymer cement in general exhibits an increase in compressive strength as the curing temperature and pressure is increased. On the average, composition 22 yields a compressive strength of 3000 psi after 24 hours of curing at temperature and pressure ranging from 60-100 °C and 1000-3000 psi. This trend supports most of the previous findings which states that compressive strength of geopolymer cement is a function of pressure and temperature[2, 7, 18-20].

CONCLUSIONS

A total of 36 formulations with different compositions of alkali concentration, fly ash to alkali binder ratio and sodium silicate to sodium hydroxide ratio were tested according to the API-10B standard testing procedures for slurry rheology and density, fluid loss, thickening time and compressive strength. Based on the experiment conducted on the properties of the class F fly ash based geopolymer cement, it can be concluded that 12M molarity of NaOH, fly ash to alkali binder ratio of 60:30, Na_2SiO_3 to NaOH ratio of 0.5, total water content of 10% of the total slurry mass and 10 ml of dispersant is the optimum composition for oilwell cementing applications. The optimum identified formulation yields a slurry mixture with a density of 15.8 ppg, plastic viscosity of 127.5 cP, yield point of 12.5 lb/ft^2, fluid loss of 56 ml in 30 minutes and consistency of 82 Bc after 4 hours which meets the general industry requirements for oil well cementing purposes. Moreover, the identified composition yields a compressive strength of 3000 psi after 24 hours of curing at temperature and pressure ranging from 60-100 °C and 1000-3000psi. This further suggests that class F fly ash based geopolymer cement could be a better replacement for the currently used OPC cement for oil well cementing applications at high pressure and temperature conditions.

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