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Mechanical Properties of Different Alkali Activated Slag Content for Oilwell Cement under Elevated Conditions

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Abstract

The increase in awareness towards global warming has prompted the research of alternatives to the conventional ordinary Portland Cement (OPC). The Class G OPC produces huge amounts of carbon dioxide (CO2) gas during its production. The slag based geopolymer cement has been identified as a potential greener alternative. Slag is chosen as the raw material because it contains silica and aluminum which is classified as pozzolan material that can be used to synthesis geopolymer cement. As most of the research done for slag based geopolymer cement is for the construction industry, and therefore, the parameters, ratios and compositions of the cement need to be improved to suit oilwell cementing criteria. The cement was tested for rheology test, fluid loss test and compressive strength test according to API cement testing procedures. The results shown that, 20% volume of water, in relative to volume of slag and alkali activator produced the optimum density and viscosity result compared to 10% and 30% of water. Meanwhile, Sodium Hydroxide (NaOH) to Sodium Silicate (Na_2SiO_3) ratio of 1:2.5 gave the ideal result compared to the ratio of 1:0.5, 1:1 and 1:2. Optimum density and viscosity were achieved using NaOH molarity of 12M instead of 10M and 14M. When the slurry with different volume of dispersant were kept for 10 minutes, the thickening time varied. The optimum dispersant volume was 10mL which enabled the slurry to remain in liquid state at the end of the 10 minute test duration.

Keywords: slag, geopolymer cement, oilwell cement, elevated conditions

1 Introduction

It is shown that, approximately one ton of CO_2 is released to the atmosphere for the production of one ton of Portland cement [1]. Due to the increasing awareness in addressing this issue, viable replacement for the conventional Portland cement is currently being reviewed and studied in detail. Recent studies show 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]. Therefore there is a dire need to develop a sustainable cement technology which possesses superior properties compared to the conventional Portland cement for oil well cementing. This research focuses on the potential of slag based geopolymer cement for the oilwell cementing applications.

2 Slag based Geopolymer Cement

Geopolymer cement is an inorganic binder which can be polymerized from materials which are rich in silica and alumina. Joseph Davidovits (1970), a renowned French scientist and engineer, first introduced the term “geopolymer” by synthesizing a reaction between alumina silicate powders with an alkaline solution. As compared to the conventional Portland cement, the geopolymer cement significantly reduces the emission of CO_2 without compromising the overall cement performance in an array of applications [3]. The geopolymerization process can be described as the geosynthesis which incorporates naturally occurring silico-aluminates. 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. On the whole, the process of geopolymerization involves the rapid chemical reaction in an alkaline environment on Si-Al minerals. The geosynthesis of geopolymer would greatly depend on the ability aluminum ion to initiate chemical changes in the silica backbone [4]. 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 will yield in different geopolymer strength and properties associated with it. There are few types of geopolymer cement, namely slag based, rock based and fly ash based. The slag based geopolymer cement is discussed in detail since this research focuses on the potential of slag based geopolymer cement for the oilwell cementing applications.

Pozzolans are any fine material which contains silica and alumina. In its natural form, pozzolans does not present any cementing characteristics, however it displays the cementing characteristics in the presence of calcium oxide and calcium hydroxide. Slag and fly ash are some examples of pozzolans. Slag is a bi-product formed during the manufacture of iron from iron ore whereby it is a residual from coke and fluxes combustion. According to Zhang Y, et al. [5] slag consists of SiO_2 and Al_2O_3 which enables them to be clustered under pozzolan materials. Besides that, it was found that by adding slag, higher strength of the geopolymer cement can be achieved because slag delivers more calcium to the structure [6]. Moreover, studies have shown that, the production of slag emits 80% lower greenhouse gases compared to 1 ton of CO_2 emitted to the environment through OPC production [7].

Formulation of Slag based Geopolymer Cement

Due to the lack of experimental work performed in the area of slag based geopolymer cement, the optimum formulation used to cure fly ash based geopolymer cement was used as a basis of designing this experiment. Alkaline solution was used for the geopolymerization process of the cement. The most commonly used alkaline solution is the combination of NaOH solution and Na_2SiO_3 solution. Joshi S.V. & Kadu M.S. [8] suggested that the ratio of 1:2.5 of NaOH to Na_2SiO_3 gives the highest compressive strength for fly ash based geopolymer compared to the ratio of 1:1.75, 1:2, 1:2.25, and 1:3. In a study conducted by Joe, M et al.[9] the fly ash based geopolymer cement which synthesize using NaOH with the molarity of 12M gave the highest compressive strength compared to NaOH molarity of 8M, 10M, 14M and 16M. Based on the experiment the fly ash geopolymer cement specimen with 12M NaOH gave 1.25 times higher compressive strength compared to other molarities. In a study performed by Panda et al. [10] it was found that the compressive strength of fly ash increased with the addition of Na_2SiO_3. In this study, the quantity of water is defined as the total sum of water contained in the NaOH, Na_2SiO_3 activator and also the added water. Meanwhile the quantity of solid is the total mass of the slag and the solid present in the alkaline activators. The effect of adding dispersants, which is used to improve the rheological properties of the cement slurry is also examined.

Nasvi et.al [11] stated that the curing temperature for fly ash based geopolymer cement is 30-80°C. This information if further verified through the experiment which proves that both class G and also geopolymer cement gains compressive strength as the temperature increases. The study also showed that geopolymer cement possesses the highest compressive strength which is 87.5MPa at 80°C. This is because at high temperature the reaction rate increases and the mechanical strength also increases. In addition, the study also showed that the curing temperature should not be more that 100°C as this could break the intergranular structure of the geopolymer and lead to strength reduction. Meanwhile Sugumaran M [12] found that longer curing time increases the compressive strength of geopolymer cement.

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. A study performed by Joshi S.V. & Kadu M.S. [8] showed that the curing time of 24 hours and 48 hours does not influence the compressive strength of the geopolymer cement rapidly. Thus, curing for 24 hours is adequate. Moreover curing for 24 hours is applicable for oilwell applications as longer curing time results in more spending and also possibilities of lost circulations.

3 Experimental Methodology

Several experiments were designed and conducted according to the API cement testing procedures. The total volume of cement slurry for all the cases were kept constant at 600mL. NaOH pellets were diluted with deionized water. Na_2SiO_3 was then added into the NaOH solution, followed by slag and 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 was in the range of 12.5ppg to 16ppg. After the slurry tested for viscosity, it was transferred into a viscometer to determine the plastic viscosity and yield point. 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. Once the slurry passed the density and viscosity test, it was then tested for fluid loss using a fluid loss tester. The fluid loss was in the range of 100 to 200 mL in 30 minutes.

Final test was performed after the slurry passed the predetermined test. Cement sample was transferred into the curing molds of 50mmx50mmx50mm, cured at 80°C and 2000psi for 24 hours. The cured cement cubes was removed from the curing chamber after 24 hours for the compressive strength testing.

4 Results and Discussion

Rheology Test

The first parameter tested was the ratio of water to slag while maintaining the alkali activator as the independent variable. NaOH molarity was set to 12M and NaOH to Na_2SiO_3 ratio was set to 1:2.5. The water amount of 10%, 20%, 30% and 40% percent were tested in relative to the amount of slag and alkali.

The results of the experiment conducted for Case 1 is summarized in Table 1.When the cement slurry was mixed with 10% of water it was found that the cement slurry hardened while mixing in the cement mixer. This is because the there was insufficient alkali in the mixture to react with the excess slag present. The similar observation was observed whereby the cement hardened before mixing due to insufficient alkali content.

Table 1. Case 1: Cement slurry with 10% of water

Water 10% :60mL		
Slag: Alkali (60:30)	**Slag: Alkali (45:45)**	**Slag: Alkali (30:60)**
slag : 360g	slag : 270g	slag : 180g
Alkali : 180g	Alkali : 270g	Alkali : 360g
NaOH : 52g	NaOH : 77g	NaOH : 103g
Na_2SiO_3 : 128g	Na_2SiO_3 : 193g	Na_2SiO_3 : 257g
Density :*	Density :*	Density :*
300 rpm :*	300 rpm :*	300 rpm :*
600 rpm :*	600 rpm :*	600 rpm :*
200 rpm :*	200 rpm :*	200 rpm :*
100 rpm :*	100 rpm :*	100 rpm :*
6 rpm :*	6 rpm :*	6 rpm :*
3 rpm :*	3 rpm :*	3 rpm :*
PV :*	PV :*	PV :*
YP :*	YP :*	YP :*
mud rheology & density : FAIL	mud rheology & density : FAIL	mud rheology & density :FAIL

* The cement slurry hardened while mixing. No readings could be taken.

The results of the experiment conducted for Case 2 is summarized in Table 2. The sample was hardened while mixing due to insufficient amount of water (120mL) for 360g of slag. Meanwhile the cement slurry of 40% of slag, 40% of alkali and 20% of water gave the optimum result. The density was 14.1ppg which is in the range of 12.5ppg to 16ppg and the yield point was 8. Thus, this slag based geopolymer cement formulation passes the rheology test. The formulation with 20% of slag, 60% of alkali and 20% of water did not harden while mixing. However the density of the slurry, 12.2ppg was lesser than the required range of 12.5ppg to 16ppg that is practiced in oilwell cementing. Moreover the yield point was also less than 5, which is below the recommended requirement. It can be concluded that the excess water reduces the density and viscosity of the slag based geopolymer cement.

Table 2. Case 2: Cement slurry with 20% of water.

Water 20% :120mL					
Slag: Alkali (60:20)		**Slag: Alkali (40:40)**		**Slag: Alkali (20:60)**	
slag	: 360g	slag	: 240g	slag	: 120g
Alkali	: 120g	Alkali	: 240g	Alkali	: 360g
NaOH	: 34g	NaOH	: 69g	NaOH	: 103g
Na_2SiO_3	: 86g	Na_2SiO_3	: 172g	Na_2SiO_3	: 257g
Density	:*	Density	:14.1ppg	Density	:12.8ppg
300 rpm	:*	600 rpm	:82	600 rpm	:54
600 rpm	:*	300 rpm	:44	300 rpm	:29
200 rpm	:*	200 rpm	:28	200 rpm	:20
100 rpm	:*	100 rpm	:15	100 rpm	:10
6 rpm	:*	6 rpm	:2	6 rpm	:1.5
3 rpm	:*	3 rpm	:1	3 rpm	:1
PV	:*	PV	:46	PV	:25
YP	:*	YP	:8	YP	:3
mud rheology & density	: FAIL	mud rheology & density	: PASS	mud rheology & density	: FAIL

* The cement slurry hardened while mixing. No readings could be taken.

In the case for the formulation with 50% of slag, 20% of alkali and 30% of water the slurry fails the rheology test as the density is below than 12.5ppg (Table 3). Meanwhile for 35% of slag and 35% of alkali and 20% of slag and 50% of alkali the density of the slurry is below than 12.5ppg. Thus it failed the rheology test. The density fails for all the formulation with 30% of water because 30% of water is in excess for the amount of slag used. As the optimum amount of water is 20% for 240g of slag, usage of 30% water for 300g, 210g and 120g of slag is definitely in excess. Thus this water dilutes the formulation further and decreases the density and viscosity of the formulation. Hence it can be concluded that, 20% of water added to the 40% of slag and 40% alkali activator is the optimum formulation which gives the best results in the rheology test.

The next parameter which tested was the ratio of NaOH solution to Na_2SiO_3. 20% of water was used for all the test as it was identified to be optimum volume of water from the previous experiment. The molarity of NaOH was maintained at the optimum value of 12M according to the research done by Joe, M et al.[9] using fly ash which resulted in the highest compressive strength. In this experiment the ratios of NaOH to Na_2SiO_3 tested was1:0.5, 1:1, 1:2 and 1:2.5. The results of slurry formulation with varied alkali ratios is summarized in Table 4.

Table 3. Case 3: Cement slurry with 30% of water

Water 30% :180mL		
Slag: Alkali (50:20)	**Slag: Alkali (35:35)**	**Slag: Alkali (20:50)**
slag : 300g Alkali : 120g NaOH : 34g Na_2SiO_3 : 86g Density :12.3ppg 600 rpm :77 300 rpm : 40 200 rpm : 27 100 rpm : 14 6 rpm : 2 3 rpm : 1 PV : 26 YP : 2 mud rheology & density : FAIL	slag : 210g Alkali : 210g NaOH : 60g Na_2SiO_3 : 150g Density :12.1ppg 300 rpm :65 600 rpm :35 200 rpm :20 100 rpm :11 6 rpm :1.5 3 rpm :1 PV :22 YP :2 mud rheology & density :FAIL	slag : 120g Alkali : 300g NaOH : 86g Na_2SiO_3 : 214g Density :11.9ppg 300 rpm :51 600 rpm :31 200 rpm :17 100 rpm :10 6 rpm :1 3 rpm :0.5 PV :20 YP :0.5 mud rheology & density :FAIL

Table 4. Slurry formulation with varied alkali ratio

$NaOH:Na_2SiO_3$ (1:0.5)	**$NaOH:Na_2SiO_3$ (1:1)**	**$NaOH:Na_2SiO_3$ (1:2)**	**$NaOH:Na_2SiO_3$ (1:2.5)**
slag : 240g Alkali : 240g NaOH : 160g Na_2SiO_3 : 80g Density :* 300 rpm :* 600 rpm :* 200 rpm :* 100 rpm :* 6 rpm :* 3 rpm :* PV :* YP :* mud rheology & density : FAIL	slag : 40g Alkali : 40g NaOH :120g Na_2SiO_3 :120g Density :* 300 rpm :* 600 rpm :* 200 rpm :* 100 rpm :* 6 rpm :* 3 rpm :* PV :* YP :* mud rheology & density : FAIL	slag : 240g Alkali : 240g NaOH : 80g Na_2SiO_3 : 160g Density : 12.5 600 rpm : 82 300 rpm : 44 200 rpm : 29 100 rpm : 15 6 rpm : 1 3 rpm : 0.5 PV : 26 YP : 6 mud rheology & density : PASS	slag : 240g Alkali : 240g NaOH : 69g Na_2SiO_3 : 172g Density :14.1 600 rpm :82 300 rpm :44 200 rpm :28 100 rpm :15 6 rpm :2 3 rpm :1 PV :46 YP :8 mud rheology & density : PASS

* The cement slurry hardened while mixing. No readings could be taken.

Among the four ratios, the slurry with the ratio of 1:0.5 and 1:1 failed the rheological test as both slurries hardened before the density and viscosity test were carried out. This is because the amount of Na_2SiO_3 is less than NaOH. The excess NaOH molecules will bind to the free water and cause deficiency of free water to dilute the slag. Meanwhile, NaOH: Na_2SiO_3 ratio of 1:2 and 1:2.5 demonstrated reasonable rheology properties. The density of the slurry for both of these slurries were in the required range of 12.5ppg to 14.1ppg. However the alkali ratio of 1:2.5 resulted in higher plastic viscosity (PV) and yield point (YP). Hence the slurry with NaOH to Na_2SiO_3 ratio of 1:2.5 is recommended in terms of rheological properties. However, the ratio of 1:2.0 can still be considered as the rheology test results falls within the required range.

Table 5. Slurry formulation with varied sodium hydroxide molarity.

NaOH molarity 10M (28gpellets)	**NaOH molarity 12M (33gpellets)**	**NaOH molarity 14M (39gpellets)**
slag : 240g	slag : 240g	slag : 240g
Alkali : 240g	Alkali : 240g	Alkali : 240g
NaOH : 69g	NaOH : 69g	NaOH : 69g
Na_2SiO_3 : 172g	Na_2SiO_3 : 172g	Na_2SiO_3 : 172g
Density : *	Density :14.1ppg	density :12.8ppg
600 rpm : *	600 rpm :82	600 rpm :73
300 rpm : *	300 rpm :44	300 rpm :38
200 rpm : *	200 rpm :28	200 rpm :26
100 rpm : *	100 rpm :15	100 rpm :13
6 rpm : *	6 rpm :2	6 rpm :4
3 rpm : *	3 rpm :1	3 rpm :1
PV : *	PV :46	PV :20
YP : *	YP :8	YP :3
3' rpm : *	3' rpm :1	3' rpm :0.5
mud rheology & density : FAIL	mud rheology & density : PASS	mud rheology & density : FAIL

* The cement slurry hardens while mixing. No readings could be taken.

The third parameter which was tested is the NaOH molarity. Joe, M et al.[9] using fly ash based geopolymer, the NaOH molarity of 12M was the optimum compared to 8M, 14M and 16M. In this research, experiments were carried out to identify the optimum NaOH molarity using slag based geopolymer which suits oilwell cementing applications. 20% of water and 1:2.5 ratio of NaOH to Na_2SiO_3 were used in all the test in this experiment, as it was found to be optimum range from the previous experiments.

The results of slurry formulation with varied NaOH molarity is summarized in Table 5. In this experiment 3 different slurries with different NaOH molarities were prepared namely 10M, 12M and 14M. Among the three slurries, the cement slurry formulation with 12M gave the optimum result. The density and the viscosity of this slurry was within the required range. However when lower molarity of NaOH was used (10M) the slurry thickened while mixing. This is due to the insufficient amounts of NaOH molecules to bind with Na_2SiO_3 molecules. However, when 14M NaOH was tested, the density of the slurry reduces. This phenomena can be attributed to the excess of NaOH molecules in relative to Na_2SiO_3 molecules. The excess NaOH results in additional water in the slurry which reduces viscosity and density. In conclusion 12M of NaOH is the optimum molarity for the 40% slag based geopolymer cement.

Table 6. Slurry formulation with varied dispersant volume.

Dispersant volume (5mL)		Dispersant amount: (10mL)		Dispersant volume (15mL)		Dispersant amount (20mL)	
slag	: 240g	slag	: 240g	slag	: 240g	slag	: 240g
Alkali	: 240g	Alkali	: 240g	Alkali	: 240g	Alkali	: 240g
NaOH	: 69g	NaOH	: 69g	NaOH	: 69g	NaOH	: 69g
Na_2SiO_3	: 172g	Na_2SiO_3	: 172g	Na_2SiO_3	: 172g	Na_2SiO_3	: 172g
Density	:14ppg	Density	:14.1ppg	Density	13.9ppg	Density	:14ppg
600 rpm	:75	600 rpm	:82	600 rpm	:86	600 rpm	:90
300 rpm	:41	300 rpm	:44	300 rpm	:47	300 rpm	:50
200 rpm	:27	200 rpm	:28	200 rpm	:31	200 rpm	:33
100 rpm	:14	100 rpm	:15	100 rpm	:16	100 rpm	:11
6 rpm	:3	6 rpm	:2	6 rpm	:2	6 rpm	:2
3 rpm	:1	3 rpm	:1	3 rpm	:1	3 rpm	:1
PV	:34	PV	:46	PV	:47	PV	:44
YP	:7	YP	:8	YP	:8	YP	:7
3' rpm	:*	3' rpm	:5	3' rpm	:1.5	3' rpm	:*
3” rpm	:*	3” rpm	:1	3” rpm	:*	3” rpm	:*
mud rheology & density	: PASS	mud rheology & density	: PASS	mud rheology & density	: PASS	mud rheology & density	: PASS

* The cement slurry hardens while mixing. No readings could be taken.

The next parameter tested was the dispersant volume. The volume of dispersant tested were 5mL, 10mL and 20mL. As per the findings in the earlier tests, 20% of water, NaOH to Na_2SiO_3 ratio of 1:2.5 was used and NaOH molarity was set to 12M.

The results of slurry formulation with varied dispersant volume is summarized in Table 6. In this test the formulation with 20% of water, 1:2.5 alkali ratio and 12M of NaOH was tested with different volumes of dispersant. The cement slurry added with 10mL of dispersant gave the optimum result. When the volume of dispersant is reduced to 5mL, the cement slurry hardened while mixing. This is because the volume of dispersant is insufficient to disperse the volume of slurry efficiently. Meanwhile when the volume of dispersant was increased to 15 and 20mL, the cement slurry took longer to solidify. The excess dispersant binds with the alkali solution and disperses the alkali solution. This prevents the alkali molecules from binding with the slag particles and causes the cement slurry to harden quickly. Thus 10mL of dispersant is the optimum volume of dispersant for 40% of slag.

In conclusion there were two formulation of cement which passed the rheology test. As tabulated in Table 7, the difference between these two formulations are the NaOH to Na_2SiO_3 ratio. Since both ratios of 1:2 and 1:2.5 passed the rheology test, fluid loss test would be performed on both of this formulation.

Table 7. Formulation which passed the rheology test.

Sample	Slag to alkali ratio	Volume of water	NaOH: Na_2SiO_3	NaOH molarity(M)	Volume of dispersant(mL)
1	40:40	20%	1:2.0	12	10
2	40:40	20%	1:2.5	12	10

Fluid Loss Test

The fluid loss test were conducted on both the formulation of slag based geopolymer cement which passed the rheology test. The test was conducted at 1500psi and 250°F. The result of the fluid loss test is summarized in the Table 8.

Table 8. Fluid loss result.

Sample	Time taken (min)	Fluid loss (mL)	Remark
Sample 1	30	25	Pass
Sample 2	30	7	Pass

According to the API specification the fluid loss characteristics for class G cement ranges from 75mL to 100mL in thirty (30) minutes. Based on the results from this experiment, as tabulated in Table 8, the fluid loss obtained was lesser than the required API specifications. In addition. API specification states that if the fluid loss is less than 50mL it can be used for tight gas formation. The fluid loss recorded for slag based geopolymer cement is lower because, slag on its own possess cementitious characteristics.

Compressive Strength Test

The compressive strength test were conducted for both samples which passed the rheology test. Based on the literature review on fly ash based geopolymer [12], the compressive strength increases when Na_2SiO_3 concentration increases. In this research using slag based geopolymer, Sample 2 has higher concentration of Na_2SiO_3 compared to Sample 1. The cement slurries were cured for 24 hours at pressure of 2000 psi and temperature of 80°C. Then the cured cement molds were left in water bath for one (1) hour before it was tested for compressive strength. The compressive strength result for both of the samples are summarized in the Table 9.

Table 9. Compressive strength test result.

Sample	Compressive strength (kN)	Compressive strength (psi)
Sample 1	36.5	3650
Sample 2	51.1	5110

The result obtained from the compressive strength test conforms to the literature study [12] where the compressive strength of geopolymer cement increases as the concentration of Na_2SiO_3 increases. Sample 2 with highest Na_2SiO_3 amount gives the highest compressive strength.

5 Conclusions

Based on the experiment conducted on the properties of the slag based geopolymer cement, it can be concluded that 20% volume of water, with 1:2.5 ratio of NaOH to Na_2SiO_3, 12M NaOH and 10mL volume of dispersant is the optimum composition for oilwell cementing applications. This formulation of slag based geopolymer cement is also suitable for tight gas formation because it possesses lower fluid loss in comparison to the conventional class G cement. In addition, the compressive strength of the slag based geopolymer cement increases with the increase in the concentration of Na_2SiO_3 .

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