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Formulation of geopolymer cement using mixture of slag and class f fly ash for oil well cementing

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Abstract. The increase in greenhouse gas emissions has been a factor for the increase in global temperature. Geopolymer cement has been intensively studied to replace conventional ordinary Portland cement, however the focus is limited to civil purposes under atmospheric conditions. This research focuses on the formulation of geopolymer cement to be used in oil well cementing application by taking account the effect of sodium hydroxide (NaoH) molarity, ratio of alkali binder and fly ash, amount of dispersant for oilwell operation under temperature ranging of 80˚C and 90C˚ and pressure of 1000 and 3000psi. The formulated composition is tested for fluid loss where the standard has been from 60 to 80 ml. The cement slurry is cured in a 50mm x 50mm x 50mm mold for period of 24 hours. Four manipulating variables were set in formulating the cement slurry namely, the ratio between fly ash and slag to alkali binder, ratio of sodium hydroxide (NaoH) to sodium silicate, molarity of NaoH and amount of dispersant added. After running a set of 16 experiment, sample (12) was found to possess the best rheological properties and fluid loss according to API RP10B. It was found that as the curing temperature and pressure increase, the compressive strength of the formulated geopolymer cement also increased.

1. Introduction

Cementing is a fundamental and important process in drilling a well. Oil well cementing provides zonal isolation and support to the casing in the wellbore. Cementing is done after the casing is placed in the wellbore, whereby cement slurry is pumped through the drill pipe into the annulus to fill the empty space between the casing and the wall of the formation. Cement slurry design is very much dependent on the depth and environment of the well placement. The design of cementing job for wells located deep in the reservoir is very much a challenge due to high temperature, pressure and the presence of corrosive fluids.

Typically, Ordinary Portland Cement (OPC) is used to form the cement slurry around the globe, but studies have shown that the OPC tends to degrade over the time due to chemical reaction which takes place in acidic wellbore environment [1][2]. Besides that, OPC also shows the trend of decreasing yield strength in application in high pressure and temperature wells [3]. It was found that, approximately one ton of CO_2 is released to the atmosphere for the production of one ton of OPC [4]. Due to the increasing awareness in addressing this issue, viable replacement for the conventional Portland cement is currently being reviewed and studied in detail. Geopolymer cement is one of the emerging alternative to replace the OPC in all construction and industrial processes. Increased strength and durability in shorter curing time is the key attribute of Geopolymer cement compared to OPC [5].

Geopolymer cement can be formulated using various cementitious component such as fly ash, slag, rock and ferro-sialate based materials, making it very much viable to form instead of relying throughout on calcium carbonate rocks as OPC. Geopolymer cement also shows superior qualities of being more resistant to acidic wellbore environment compared to OPC due to its low calcium carbonate content [6].

2. Methodology

The methodology was divided into four parts. The first part of this experiment was the formulation of geopolymer cement. The variables in formulating the cement slurry were the ratio of slag and fly ash to alkali binder, the ratio between Sodium Hydroxide and Sodium Silicate, the molarity of Sodium Hydroxide and the amount of dispersant added to cement slurry.

2.1. Geopolymer cement formulation

The ratio between Sodium Hydroxide and Sodium Silicate was set to constant of 1:1 throughout the experiment. Sodium Hydroxide and Sodium Silicate was used as alkali binder for this experiment, the ratio between Sodium Hydroxide and Sodium Silicate changed and analysed, molarity of sodium hydroxide was also varied. The molarity used for this experiment was 10, 12 and 14 mol/L. Other factors analysed was the amount of dispersant added to the cement slurry whereby the amount was varied between 3,6,10 and 12ml. Fly ash, slag, Sodium Hydroxide and Sodium Silicate is then mixed using the speed mixer at 1200 rpm for duration of one minute with the addition of dispersant. To increase the thickening time of the cement slurry, water (20% of total weight of the mixture) was also added to the mixture. In accordance to API standard, the cement slurry was designed according to parameter listed in Table 1.

Table1: API Cement Slurry Rheology

Test Parameter	Industry standard
Density	12.5 – 16 ppg
Viscometer	PV> Mud Cp and YP>5
Compressive strength	>500psi

2.2. Fluid Rheology and density test

The formulated mixture was tested for its fluid rheological properties using the viscometer to determine if the mixture adheres to the API standard. The test is carried out using Fann300 viscometer. Reading were taken after the viscometer is run for 5 seconds. The stabilised reading at 600rpm and 300rpm was taken respectively. The readings were used to calculate the Plastic Viscosity (PV) and Yield Point (YP) of the cement slurry.

2.3. Fluid Loss Test procedure

Fluid loss test was performed to identify the amount of fluid diffused out of the cement slurry to adjacent formation at high pressure and temperature. This test was performed using static fluid loss tester at pressure of 1000psi and temperature of 260 °C. Measuring cylinder is placed below the equipment after the pressure is applied to cement slurry. The time also observed using stopwatch. After the period of 30 minutes, the pressure was removed and the total amount of water loss was recorded.

2.4. Compressive Strength test

Cement cubes were obtained from the cement mold after the curing process. The dimensions of the cement cubic was measured, and the surface area of the cubic was calculated. The cement cubic was then loaded in the compressive strength tester and the load was increased uniformly. The force where the cement cube fails was noted and recorded as ‘Fi’. The compressive strength of the cement cube was calculated using Equation (1).

$$\sigma i = \frac{Fi}{Ai} \ldots\ldots\ldots\ldots\ldots\ldots\ldots \text{ Equation (1)}$$

Where;
σi= compressive strength, N/$mm2$
Fi= maximum load, N
Ai= cross-section area

3. Results and Discussion

3.1. Cement Slurry Rheology and density

After the formulation of each cement slurry, it was tested for its rheological properties and density. The formulation that matches the API standards was further tested with fluid loss test and compressive strength test, meanwhile other formulation were considered as failed. After formulating 12 geopolymer cement slurries, sample (12) was chosen as the best cement slurry which met the API standard requirements. The composition and rheological test of sample (12) is listed in Table 2 and 3.

Table 2: Composition of sample (12)

Total weight (g)	600
H20 (g)	120
FA(g)	150
Slag(g)	150
Sodium Hydroxide (g)	60
Sodium Silicate (g)	120
Dispersant (g)	10

Table 3 : Density and Rheology test result sample (12)

Density	13.4
Plastic viscosity	31
Yield point	6

The results show that composition of slag and fly ash mixture to alkali binder affects the fluid rheological properties. In the second study, the ratio between sodium hydroxide and sodium silicate was varied. It was identified that the ratio of 1:05 produces the best results in fluid rheological test. Addition of Sodium Silicate increases the viscosity of the cement slurry. However above the optimum amount of Sodium Silicate, further addition leads to gelling effect of the cement which eventually fails in the PV and YP test. For the molarity of the Sodium Hydroxide solution, the molarity of 12 mol/litre^3 gave better results compared to the 14 mol/litre^3 and 10 mol/litre^3. When running the experiment with 10 mol/litre^3 and 14 mol/litre^3 the samples failed in PV and YP test. The effect of dispersant was also analysed, whereby it was found that the addition of dispersants reduces the gelling effect of the cement slurry. Above the optimum amount, further addition causes the failure of cement slurry in PV and YP test meanwhile below the optimum amount it leads to the gelling of cement slurry.

3.2. Compressive Strength test

Cement mold of sample (12) was cured at different temperatures and pressures in a 50mm x 50mm x 50mm cement mold. The compressive strength test was carried out using the destructive compressive strength tester to investigate the mechanical behaviour under high temperature and pressure. The results of compressive strength of the cement molds are listed in Table 4.

Table 4: Test 1 and Test 2 results

Test 1	
Reading of compressive strength tester	50.8 kN
Curing pressure	1000 psi
Curing temperature	80° Celsius
Compressive strength	2946.4psi
Test 2	
Reading of compressive strength tester	79.7 kN
Curing pressure	3000 psi
Curing temperature	90° Celsius
Compressive strength	4622.6 psi

The increase in curing temperature and pressure resulted in an increase in the compressive strength of the formulated geopolymer cement. This is in contrast with OPC where the compressive strength decreases as curing temperature and pressure increases [7].

4. Conclusion

In this research on geopolymer cement formulated using mixture of Class C Fly Ash and slag, it was found that geopolymer cement could be a better replacement to the currently used OPC cement for oil well cementing applications in high pressure and temperature wellbore environment. It's also shown in this study that the compressive strength of geopolymer cement does not show a steep decrease compared to OPC in high pressure and temperature, instead it increases as the curing temperature and pressure is increased. In addition, this study has also identified the factors which determines the rheological properties of cement slurry to match the standard required by industry. However, to be applied in the oil well cementing industrial, further studies on the additives should be conducted to give an ideal cement slurry with improved compressive strength.

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