Elasticity and expansion test performance of geopolymer as oil well cement

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Abstract. History has shown that geopolymer cement provides high compressive strength as compared to Class G cement. However, the research had been done at ambient temperature, not at elevated condition which is the common oil well situation. In this research, the physical and mechanical properties performance of the oil well cement were investigated by laboratory work for two types of cement that are geopolymer and Class G cement. The cement samples were produced by mixing the cement according to the API standards. Class C fly ash was used in this study. The alkaline solution was prepared by mixing sodium silicate with NaOH solution. The NaOH solution was prepared by diluting NaOH pellets with distilled water to 8M. The cement samples were cured at a pressure of 3000 psi and a temperature of 130 °C to simulate the downhole condition. After curing, the physical properties of the cement samples were investigated using OYO Sonic Viewer to determine their elastic properties. Autoclave expansion test and compressive strength tests were conducted to determine the expansion value and the strength of the cement samples, respectively. The results showed that the geopolymer cement has a better physical and mechanical properties as compared with Class G cement at elevated condition.

1. Introduction

Well cementing process is one of the most crucial steps in drilling and well completion phase. In ensuring a good zonal isolation, great wellbore stability, corrosion control and to avoid the invasion of formation fluid into the wellbore, a good cementing process is needed. Well cementing is an act of placing cement sheath in between casing and formation [1]. It is a crucial aspect in well completion whereby good cementing job ensures the longer life of cement thus protects the wellbore integrity as long as the production is going on. While, in abandonment phase, the cement plugging is important to shut the pressure and maintaining the reservoir pressure at a safe condition. Thus, its physical and mechanical properties when exposed under high pressure and high temperature condition need to be investigated.

In current industrial practices, Ordinary Portland Cement (OPC) specifically Class G cement is the common type of cement being used. However, there is a newly fly ash-based geopolymer cement is being evaluated in order to replace the usage of Class G cement as it offer higher mechanical strength, high durability and low shrinkage as compared to the Class G cement [2-3].

Nordin et al. [3] emphasized that the utilization of fly ash in building development had a great track record and it is presently utilized in the United States. However, many design experts proceed to remain overly restrictive with regards to utilizing fly ash as a part of construction materials [3]. Hence, many researchers have conducted test and experiments in ensuring the geopolymer is commercially viable to be use in the industry [2, 4-7]. The chemical structure and activation between these two cements differ...
Geopolymers are synthesized by alkaline activation of aluminosilicate based materials like fly ash. Geopolymers undergo a process called geopolymization, a process involving series of dissolution, reorientation, solidification reaction. It categorizes according to its amount of physical properties in such smoothness of the surface, thermal strength, long-term sturdiness and the adhesiveness between particle and the microstructure of geopolymers is dependent on the temperature [4]. Nasvi et al. [2] mentioned that both geopolymers and Class G cement gained strength with the curing temperature up to their optimum curing temperatures which lies between 55-60 °C. Geopolymer had gained higher increment in strength as compared with Class G cement which is 92%, whereas that Class G cement is 46%. They also mentioned that there is a possibility of breaking up intergranular structure of geopolymer at very high curing temperatures (>100 °C) [2]. Furthermore, it is observed that Class G cement possesses higher strength at lower curing temperature (<36 °C), whereas geopolymer possesses the highest compressive strength at higher temperature (> 36 °C) [2].

Ryu et al. [5] had conducted a study to investigate the mechanical properties of fly ash-based geopolymer cement where it is found that this type of cement is more suitable for precast concrete products because the compressive strength of geopolymer concrete can be developed at temperatures that exceed 60 °C. While, a study by Khalifeh et al. [6] stated that the strength of geopolymer is improved gradually up to 7 days of curing when cured at 50 °C and a pressure of 2000 psi. Next, a study conducted by Suppiah [7] concluded that the geopolymer cement with 8 to 12M of sodium hydroxide concentration cured at 93 °C and a pressure of 3000 psi was good enough for zonal isolation for oil well cementing but more search is needed to be carried out for other crucial properties of the system to justify that geopolymer cement qualifies as oil well cement.

Hence, the aim of this research paper is to study and evaluate the physical and mechanical properties for both geopolymer and Class G cement in terms of Poisson’s ratio, Young’s Modulus, expansion performance and compressive strength when cured at elevated condition at a temperature of 130 °C and a pressure of 3000 psi. It is found that less extensive research was done on studying the geopolymer cement behaviour at elevated curing condition. Due to this issue, the concept on geopolymer cement performance prior to oil well application is not fully understandable.

2. Experimental Methodology

Class C fly ash was used in this study. The chemical composition of fly ash precursor is 32.44 SiO₂, 12.22% Al₂O₃, 23.58% Fe₂O₃, 20.66% CaO, 2.35% MgO, 2.40% SO₃, 0.87% Na₂O and 5.48% containing other compounds. API Class G cement was procured from Lafarge Pasir Gudang Plant, Johor. The experiment was started by preparing the cement cubes based on API RP-10A procedures by using Constant Speed Mixer [8]. Geopolymer cement slurry contained the mixture of Class C Fly Ash and alkaline solution. While, Class G cement slurry contained the mixture of API Class G cement with distilled water. Alkaline solution was prepared by mixing sodium hydroxide of 8 M and 97 wt.% sodium silicate with the ratio of 1:2.5 for geopolymer cement sample. The cement slurry was mixed at 4,000 rpm for 15 seconds and continued by increasing the speed to 12,000 rpm for another 35 seconds. The composition of 0.44 alkaline activator/ distilled water to cement ratio was used. As reported by Ridha and Yerikania [1], the cement achieved highest compressive strength when the alkaline activator-to-fly ash ratio is 0.44 [1].

Once the slurry has been prepared, the slurry of the cement samples undergone elastic experiment such as Young Modulus and Poisson’s Ratio was poured into 2 x 2 x 4-inch mould. While, the slurry of the cement samples undergone cement expansion and compressive strength tests was poured into 2 x 2 x 2-inch cubic mould. Then, the cement samples were cured inside High Pressure High Temperature (HPHT) curing chamber contain pressure and temperature of 3,000 psi and 130 °C, respectively, for 24 hours.

After curing process, the cement samples undergone elastic experiment were cored into cylindrical shaped samples or known as coring process. Core cement specimens was prepared with a minimum diameter of 1.3 inch and the height to diameter ratio was between 2 – 2.5. The elastic properties of the
cement samples were obtained using Sonic Viewer-SX by OYO Corporation. The result from the Sonic Viewer was shown in a receipt with value of Elastic Coefficient and Poisson’s ratio.

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\text{Cement Expansion} = \frac{\text{Final Reading, } L_2 - \text{Initial Reading, } L_1}{\text{Initial Reading, } L_1} \times 100\%
\]  

(1)

Next, the expansion test for the cement samples was conducted using Autoclave Expansion Test. The test was conducted using autoclave by finding the percentage of cement expansion or shrinkage using equation (1).

Lastly, the compressive strength of the cement samples was further investigated using ELE ADR 3000 with a capacity of 3,000 kN in the -inch cubical samples. The compressive strength test was measured according to ASTM C 109 [9].

3. Result

3.1. Young’s modulus and Poisson’s ratio behaviour of geopolymer and Class G cements

The Young’s Modulus and Poisson’s ratio results for geopolymer cement and Class G cement are shown in table 1 and table 2, respectively. From the results obtained, it can be seen that the Young’s modulus of geopolymer cement sample have the average value of $2.20 \times 10^6$ psi which is higher than the Class G cement sample, $1.83 \times 10^6$ psi. The percentage of difference between the two cement samples is 18.36%. The findings from this experiment is contradicted with the study conducted by Nasvi et al. [2] which stated that both geopolymer and Class G cement shows reduction in Young’s modulus values towards 80 °C. The findings from this experiment show that the Young’s modulus for both geopolymer and Class G cement that were cured at 130 °C are higher as compared to the study by [2] which was cured at 80 °C. It can be concluded that the Young’s modulus for geopolymer cement is stiffer at elevated temperatures than at lower curing temperatures, suggesting that the elevated curing conditions had caused the geopolymer cement to truly set [10, 11]. Noushini et al. [11] explained that heat curing plays a significant role in the strength development of fly ash-based geopolymer as this type of cement made of sodium silicate solution, where the main reaction product is sodium-alumino-silicate-hydrate (N-A-S-H) gel. N-A-S-H gel is the main binder that contributes to the strength of geopolymer cement [11].

Next, the Poisson’s ratio of geopolymer cement have the average value of 0.34, which is higher than the Class G cement that has an average value of 0.294. The differences obtained for these types of cements are the results of geopolymer has more elasticity as it governs the principles of materials when the force is applied, as a diameter of a material is thinner and the length is elongated more.

**Table 1.** Young's Modulus and Poisson’s Ratio data recorded for geopolymer cement sample.

| Sample | Length (inch) | Diameter (inch) | Mass (g) | Density, $\rho$ (g/cm$^3$) | Young’s Modulus (Psi) | Poisson’s Ratio |
|--------|---------------|----------------|----------|-----------------------------|-----------------------|-----------------|
| GP-1   | 2.95          | 1.57           | 206.71   | 2.20                        | $1.94 \times 10^6$    | 0.34            |
| GP-2   | 3.07          | 1.52           | 201.70   | 2.22                        | $2.20 \times 10^6$    | 0.36            |
| GP-3   | 2.98          | 1.51           | 202.68   | 2.31                        | $2.38 \times 10^6$    | 0.34            |
| GP-4   | 3.00          | 1.51           | 207.10   | 2.21                        | $2.26 \times 10^6$    | 0.35            |
| GP-5   | 2.97          | 1.42           | 200.49   | 2.21                        | $2.20 \times 10^6$    | 0.32            |
Table 2. Young's Modulus and Poisson’s Ratio data recorded for Class G cement sample.

| Sample | Sample Dimension | Elastic Properties Data |
|--------|------------------|-------------------------|
|        | Length (inch)    | Diameter (inch) | Mass (g) | Density, ρ (g/cm³) | Young’s Modulus (Psi) | Poisson’s Ratio |
| CG-1   | 3.12             | 1.56           | 186.12   | 1.90            | 1.57×10⁶              | 0.34          |
| CG-2   | 3.10             | 1.56           | 185.23   | 1.91            | 1.91×10⁶              | 0.26          |
| CG-3   | 3.11             | 1.56           | 187.91   | 1.93            | 1.91×10⁶              | 0.30          |
| CG-4   | 3.11             | 1.56           | 186.69   | 1.92            | 1.83×10⁶              | 0.32          |
| CG-5   | 3.09             | 1.55           | 184.32   | 1.91            | 1.91×10⁶              | 0.25          |

Table 3. Autoclave Expansion Test result for both geopolymer and Class G cements.

| Sample | Initial Reading, L₁ (mm) | Final Reading, L₂ (mm) | Cement Expansion (%) |
|--------|--------------------------|------------------------|----------------------|
| Geopolymer | GP-1  | 50.11                  | 49.83                | -0.56                |
| Cement  | GP-2  | 50.84                  | 50.33                | -1.01                |
|         | GP-3  | 50.92                  | 50.72                | -0.39                |
|         | GP-4  | 50.74                  | 50.46                | -0.55                |
|         | GP-5  | 50.89                  | 50.53                | -0.71                |
| Class G | CG    | 50.79                  | 50.88                | 0.18                 |

3.2. Autoclave expansion test

Table 3 shows the autoclave expansion test for both geopolymer and Class G cement. The average shrinkage value for geopolymer cement sample is 0.644% while the Class G cement has an expansion value of 0.18%. Based on the study conducted by Khadum [12], he mentioned that the expansion of all types of Portland cement must not exceed 0.80% as stated by ASTM C151-05. The results of this study are also in agreement to Horkoss et al. [13]. They found that their cement samples had an expansion value lower than 0.85 [13]. The cement expansion of geopolymer and Class G cement in this test is still within the allowable range of cement expansion.

Khadum [12] explained that the factor contributing to the cement expansion is caused by the late hydration of calcium oxide (CaO) as well as magnesium oxide (MgO) that contributes to the increase in volume after the cement had set. While Horkoss et al. [13] agreed with [12], where he mentioned that the high MgO content in the cement could generate expansion due to the hydration of periclase to brucite, more than to the formation of ettringite. The MgO in [13] is <1% while the value for MgO in this study is 2.35%, which is still low thus eliminating any risk related to brucite formation.

Kuenzel et al. [14] stated that one of the factors contributing to the shrinkage of the geopolymer cement is caused by the evaporation of water content of the cement sample. There were two types of water concentration in this study which are critical minimum water content and structural water content [14]. The critical minimum water content is defined as the water concentration of water where the cement starts to shrink and water evaporated before this concentration is considered as free water and cement shrinkage is not bound to happen [14]. While, structural water content is the water that exists in the cement after the critical minimum water content is reached and after reaching this water concentration, cement shrinkage starts to happen [14].

Curing temperature also plays important role in determining the shrinkage of geopolymer cement. The curing condition for this study was done at an elevated condition where the curing temperature is 130 °C. By performing curing at a higher temperature, the rapid geopolymerization process takes place
and this contributes about 70% of the cement strength as early as 3-4 hours of curing [15]. The shrinkage observed was low as to compare to low temperature curing and this is proved as almost all the cement shrinkage value in this study did not exceed its allowable expansion limit.

Table 4. Compressive strength behaviour of geopolymer and Class G cements.

| Sample | Compressive Strength (Psi) |
|--------|-----------------------------|
| GP-1   | 4619                        |
| GP-2   | 5508                        |
| GP-3   | 4707                        |
| GP-4   | 5053                        |
| GP-5   | 5322                        |
| CG-1   | 1188                        |
| CG-2   | 2116                        |
| CG-3   | 1810                        |
| CG-4   | 2753                        |
| CG-5   | 2596                        |

3.3. Compressive strength test
For both geopolymer and Class G cement, the curing condition is done under 24 hours curing time with the setting of elevated condition with a pressure of 3000 psi and a temperature of 130 °C. Table 4 shows the compressive strength behaviour for both geopolymer and Class G cement samples. Based on the recorded data, the compressive strength is adequate for most cementing operations which is 500 psi [16]. Thus, geopolymer offers a greater strength as compared to the conventional Class G cement.

4. Conclusion
The major aim of this project is to study the physical and mechanical properties of geopolymer as oil well cement which were cured at elevated condition. The study found that the Young’s modulus and Poisson’s ratio for geopolymer cement is higher than Class G cement as geopolymer has more elasticity. However, more studies on the elastic properties need to be conducted in the future. While, the study of expansion test performance of geopolymer cement as oil well cement is to analyze the ability of fly ash based geopolymer in terms of its expanding or shrinkage performances. Based on the testing that was conducted, all geopolymer cement sample consistently exhibit cement shrinkage properties rather than cement expansion. It is found that the factors that affecting cement shrinkage were water-to-cement ratio and curing temperature. For Class G cement, it shows consistent trend with other literature as the cement undergoes cement expansion which is the opposite of fly ash based geopolymer cement. The cement expansion was due to late hydration of late hydration of calcium oxide (CaO) as well as magnesium oxide (MgO) that contribute to the increase in volume after the cement has set. Next, it can be concluded that geopolymer cement hold a great mechanical property in terms of its compressive strength and meets the minimum required strength for cementing operation which is 500 psi for oil well cement. It is recommended that geopolymer cement substitute the usage of conventional cement as it is not only can offer such high compressive strength but also promotes to a greener environment with less CO₂ emissions.

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