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Fired-Siltstone Based Geopolymers for CO₂ Sequestration Wells

A Study on the Effect of Curing Temperature

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Abstract — Wellbore integrity during Carbon Dioxide (CO₂) storage in deep aquifers is a major problem as the currently used well cement will lose its integrity at higher temperatures and eventually leads to CO₂ leakage into the atmosphere. Researchers have conducted several studies in order to find a proper well cement material and have tested several types of geopolymers mixing fly ash and different type of cements with an alkaline solution curing in different temperatures. This paper presents a study on geopolymer composites made from fired siltstone, slag as well as alkaline liquid which were cured at temperatures ranging from 23 °C to 80 °C. Five different types of geopolymer were prepared with ratios of fired siltstone (to 700 °C) and slag as 0:100, 20:80, 30:70, 40:60 and 50:50, separately. After preparing samples with various proportions under different temperatures, the mechanical properties of them were compared. The experimental results reveal that optimum curing temperature range is 50 to 70 °C and addition of higher proportion of slag would help geopolymer gain better performance on mechanical strength compared to other geopolymer materials.

Keywords - geopolymer; curing temperature; strength; stiffness; wellbore

I. INTRODUCTION

The combustion and calcination of fossil fuels has emitted a large amount of greenhouse gases, such as carbon dioxide (CO₂) and methane (CH₄), to the atmosphere leading to adverse effects on the environment since the industrial revolution. In order to control the amount of greenhouse gases to an acceptable level, much efforts and investments have been made, and from all of those proposed approaches, CO₂ capture and storage (CCS) is considered to be one of the best solution. In the CCS process, CO₂ is captured from the discharging points such as power plants and factories and injected into underground reservoirs through borehole wells [1]. Previous studies have shown that the materials chosen for injection wells and production wells play a key role in the CO₂ injection in terms of safety, environmental impact and sustainability.

Therefore, the integrity of the wells should be made using a material, which is durable, anti-corrosive, chemically inert, adaptive to pressure and temperature variations and to avoid any leakage of CO₂. Here, to maintain the injection/production wellbore stability and to prevent the possible leakages of CO₂ or producing fluid, wellbore annular space in between formation and the casing is cemented [2]. Ordinary Portland Cement

(OPC) based well cement is the most widely used binder in primary cementing of injection and production wells worldwide. However, this OPC based well cement is susceptible to defects due to the decrease in the strength and increase of permeability and porosity of the cement. This occurs mainly due to the chemical interaction of OPC based well cement with wet CO₂, especially, super-critical CO₂ (beyond 7.38 MPa pressure and 31.8°C temperature – critical point of CO₂) at deep underground [3].

The recent studies have shown the prospective use of geopolymers as a new well cement. According to the RILEM Technical Committee 224-AAM, geopolymer materials are “essential alumino-silicates activated with alkaline solution, excluding any other alkali-activated materials that should be classified apart” [4]. Further, geopolymer is attractive from several perspectives, including the higher acid resistance characteristics, avoidance of temperature gradients, dimensional stability over a wide range of temperature (ceramic-like properties) and higher in strength and durability [2, 4]. Further, geopolymer based well cement exhibits lower permeability and shrinkage [5] while producing lower production cost and higher pump ability [6] compared to defect prone OPC based well cements. In addition, possibility of in-situ production and tailoring of the properties to produce composite materials is an added advantage in geopolymers [7]. Furthermore, the characteristics of geopolymers allows the introduction of recycled resources and mineral wastes, thus reducing the energy demand and environmental effect, which also explains why geopolymerization could be considered as an environmentally friendly and sustainable consolidation technique to come to treat aluminosilicate-based wastes, tailings and residues [7]. Because of the higher quantities of active Si and Al, waste materials like fly ash and slag has been widely used as source materials in geopolymers [2].

However, a recent study by Lahoti et al. [8] showed the ability of using alkali-silica reactive (ASR) sedimentary rock powder (siltstone & sandstone from the waste generated from excavation of rock caverns and quarries) as a resource to produce a high strength geopolymer binder. According to their results, utilization of sedimentary rock powder increases the compressive strengths of the geopolymer by 15 – 30% compared to metakaolin geopolymers [8]. Additionally, a study done by Centre for Infrastructure Engineering, Western Sydney University could convert the siltstone powder waste generated

from Eidsvold quarry, Queensland, Australia to a higher strength greener cement [9]. They used an optimum firing temperature of 550 °C and an optimum particle size of 10 µm for the siltstone powder to achieve the best pozzolanicity which increased the compressive strength of the mortar by 14% (after 7 days) with a 10% replacement of cement with siltstone powder compared to OPC mortar (water cement ratio is 0.45) [9]. Hence, it would be interesting to study the ability of fired siltstone powder as a source material for geopolymer.

Therefore, the main objective of this research study was to investigate the ability of fired siltstone as a source material for geopolymer based well cement production. The primary attention has been paid to the effect of different downhole temperatures on the strength of fired-siltstone based geopolymer cement because, a typical, well cement is exposed to diverse temperatures with a geothermal gradient of 30°C/km [10].

II. EXPERIMENTAL METHODOLOGY

A. Sample Preparation

1) Source Material

Siltstone powder (particle size <1 mm) from Eidsvold quarry, Queensland, Australia was used as the source material and the composition of the siltstone as received is shown in Table I. The siltstone was thermally treated at different temperatures (405 °C, 700 °C and 1110 °C) to obtain the optimum material for the geopolymer and the composition of the fired siltstone samples at different temperatures are showed in Table II.

The samples were kept for 24 hours under the designated temperatures in a muffle furnace to allow the possible oxidation processes of the fresh siltstone powder. Based on the mix compositions of fired siltstone in Table II, it can be noticed that the major constituents are Si and Al, hence this can be used for geopolymer production.

Geopolymer has a structure similar to an aluminosilicate glass which consist of an amorphous 3-dimensional structure [11]. In addition, the physical and chemical structures of the hardened geopolymer is affected by the silicon and aluminum ratio of the geopolymer network. When the Si/Al ratio is below 3:1, the geopolymer nets are rigid and suitable as a cement. When the Si/Al ratio increases above 3, the geopolymer net losses its rigidity and become more flexible. With higher Si/Al ratios, up to 35:1, the resultant geopolymer chains are more suited as sealants or adhesives [12]. In this study, we would only focus on Si/Al ratio below 3:1 as we are trying to develop a wellbore-cement for carbon capture and storage wells. Hence, siltstone fired at 700 °C will be good for geopolymer compared to 1110

TABLE I. COMPOSITION OF EIDSVOLD SILTSTONE AS RECEIVED

Component	Mixture proportion range (%)	Component	Mixture proportion range (%)
SiO ₂	86.7	MgO	0.09
Al ₂ O ₃	8.60	SO ₃	0.005
Fe ₂ O ₃	0.290	P ₂ O ₅	0.084
CaO	0.03	LOI	NA
K ₂ O	0.225		

TABLE II. COMPOSITION OF FIRED SILTSTONE AT DIFFERENT TEMPERATURES

Mineral name	Abundance (%)		
	405 °C	700 °C	1110 °C
Diamond (C)	19.55	16.64	10.49
TiO ₂	0.38	0.35	0.0
SiO ₂	64.98	70.03	73.37
Al ₂ Si ₂ O ₅ (OH) ₄	15.07	14.68	3.32

°C as Al₂O₃ composition is significantly reduced at 1110 °C (refer Table II).

However, for stronger geopolymer production, alkali oxides such as CaO and Fe₂O₃ are required, and these constituents are lacking in this fired siltstone. Additionally, the proportion of SiO₂ is more than what is required. Therefore, ground blast-furnace slag, an aluminosilicate-based waste was added to the fired siltstone in different proportions (such as 0:100, 20:80, 30:70, 40:60 and 50:50) to control the amount of SiO₂ and to increase alkali oxides such as CaO. This is acceptable as slag contains significant amount of CaO (refer Table III).

2) Alkaline Reactor

As mentioned above, for the geopolymerization process, the aluminosilicate source material should activate with an alkaline solution. For the present study, the alkaline liquid utilized was a mixture of 8 mol/l sodium hydroxide (NaOH) and sodium silicate (Na₂SiO₃). The ratio of NaOH to Na₂SiO₃ has been tested at different levels (1:1.5, 1:2, 1:2.5, 1:3 - by weight) in previous work and at the end 1:2 was chosen because it provides higher strength [13]. The NaOH was obtained in pellet form (98% purity from PQ Australia) with mixing 32% of NaOH pellets with 68% of water. Then this solution was left under room temperature (ambient conditions) until the excess heat was completely dissipated to avoid accelerating the setting of the geopolymers [14]. Meanwhile, the Na₂SiO₃ solution containing 44% silicate solids and 56% water was used as it is. In addition, the alkaline solution was left for 24 hours in ambient condition, prior to the geopolymer preparation, to ensure good activation of the source material in the alkaline solution.

3) Mix proportions

It was assumed that the final density of the sample would be 1800 kg/m³ [5]. The geopolymer slurry was prepared by mixing the source material (slag and fired siltstone in different proportions, such as 0:100, 20:80, 30:70, 40:60 and 50:50) and alkaline liquid in the suitable proportions. The solutions were mixed using a mechanical concrete mixer for a period of 4

TABLE III. MIX COMPOSITION IN RECEIVED SLAG

Composition	Range in slag (%)	Composition	Range in slag (%)
SiO ₂	33.0	MgO	6.0
Al ₂ O ₃	14.0	SO ₃	1.4
Fe ₂ O ₃	2.7	P ₂ O ₅	<0.1
CaO	42.0	LOI	1.45
K ₂ O	0.2		

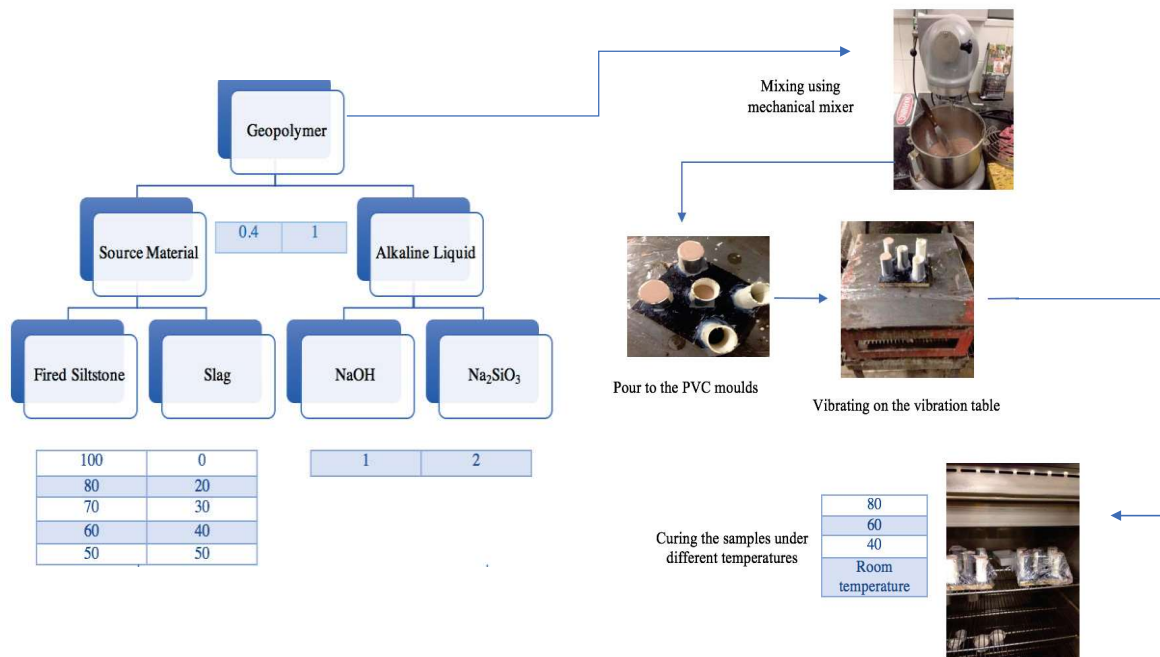


Fig. 1. Summary of the overall process of Geopolymer sample preparation using fired siltstone and slag

minutes. Based on the volume of each single specimen (for samples with 80 mm in height and 38 mm in diameter - allowing for breakages at the two edges) and the assumed density (1800 kg/m³), the weight could be calculated separately.

The weight ratio of source material to alkaline liquid used was 0.4:1, which gives the maximum compressive strength values according to previous studies [15]. To avoid the influence of experimental errors, three samples were prepared for each proportion and curing temperature, thus altogether 60 samples were prepared. The slurry was then poured into molds of 38 mm in diameter and 80 mm in height. It was poured in three layers (shown in Fig. 1) and to release any residual air bubbles, each layer was vibrated on a vibrating table for a period of around 1 minute. To prevent excessive moisture loss before curing, all molded samples were sealed with a plastic film and shifted to an air-tight container.

4) Specimen Curing

Finally, the samples were transferred to the oven (except the samples which will be cured at room temperature) and cured at different curing temperatures (40 °C, 60 °C, and 80 °C) for a

period of 24 hours. Here, these higher temperatures were used to represent the actual in situ temperature conditions expected for wellbores in deep underground (geothermal gradient of 30 °C/km [10]). After removing samples from the molds, all samples were kept at room temperature for another 72 hours prior to testing. It is acceptable as geopolymers will not develop substantial strength after 24 hours [16]. Then, the top and bottom surfaces of the specimen were machine ground by a grinder to ensure uniform axial loading during the experiment. A summary of the overall process could be seen in Fig. 1.

The geopolymer specimens made are shown in Fig. 2. It is observed that the geopolymer sample made only from fired siltstone is all white; while the slag and fired-siltstone mixed geopolymers have a grayish outer look due to impurities.

5) Specimen Testing

The compressive strength testing was conducted by Shimadzu 300 kN Uniaxial Compressive Strength testing machine (shown in Fig. 3) with a constant displacement rate of 0.2 mm/min based on strain rate for all the tests.

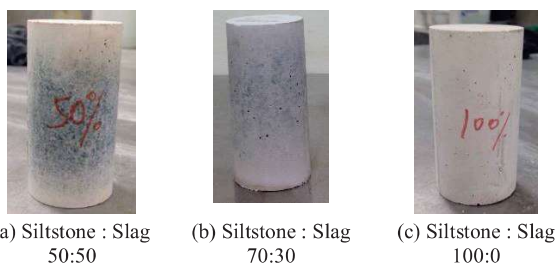


Fig. 2. Geopolymer samples after curing

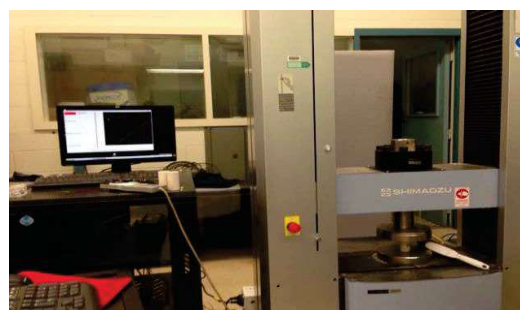
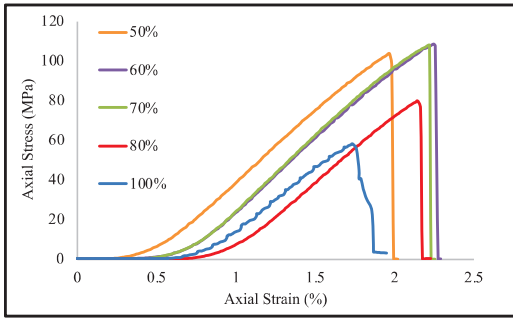
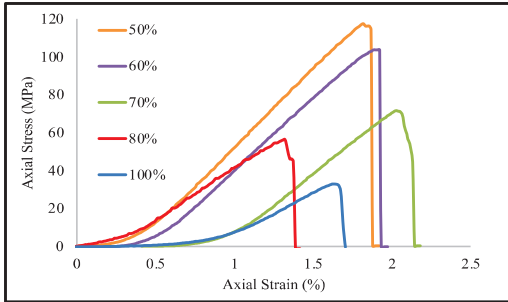


Fig. 3. Shimadzu 300KN UCS testing machine



(a) cured at room temperature



(b) cured at 60 °C

Fig. 4. Stress-strain plot

III. EXPERIMENTAL RESULTS AND DISCUSSION

A. The variation of strength with different mixing proportions and curing temperatures

1) Effect of the slag proportion

The compressive strength and axial strain development of the geopolymer cured from low temperature range (room temperature, 23 °C) up to 80 °C were investigated in this study. The stress-strain plots of the maximum compressive strength for each condition were taken for comparison and the geopolymer cured at 23 °C and 60 °C are shown in Fig. 4 separately. It is observed from the figure that the increasing of slag proportion had a positive effect on the ultimate strength.

Fig. 5 shows the compressive strength increase in geopolymer specimens with the increasing slag proportion compared to 0% slag specimen for different curing temperatures. For 20% addition of slag, the compressive strength increased by

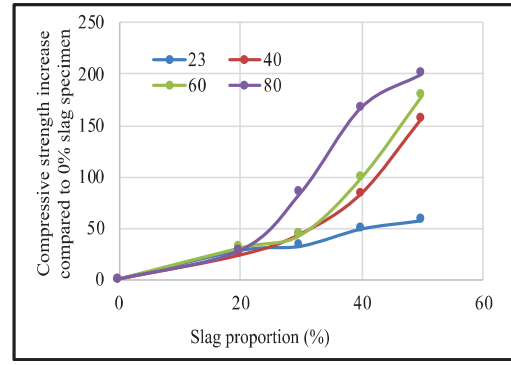







Fig. 5. Compressive strength increase of geopolymer compared to 0% of slag specimen at different curing temperatures

about 28% while it increased by about 31% when the temperature increases from 23 to 80 °C. Similar trend was observed for other slag proportions as well and the positive influence of the slag proportion further increased at higher slag percentages. For example, according to Fig. 5, 30%, 40% and 50% of slag proportions recorded around 33%, 49% and 57% of compressive strength increment for room temperature while it enhanced by approximately a factor of 1, 2 and 3 when the temperature is raised up to 60 °C respectively.

By considering the failure patterns (refer Table IV) of the specimens, it is possible to explain the observed positive influence of slag proportion in the geopolymer specimens. According to Table IV, the addition of slag has increased the stiffness and the brittleness of the geopolymer structure.

Additional slag in Si/Al ratio, has led to changes in the chemical structures and physical properties. As the slag proportion increases, the ratio of Si/Al undergoes a reduction. Based on the conclusion drawn by Guerrieri and Sanjayan [15], low ratio Si/Al provides bricks and ceramic properties to the geopolymeric materials, while a higher ratio (3:1) leads to properties similar with fiber glass composite. Figures taken when geopolymer is loaded confirmed this theory (shown in Table IV). From sample with ratio of 0:100, has components similar to fiber, which is less brittle, while sample with ratio of 50:50 share the same properties with ceramic material, which is strong in compression, brittle as well as stiff.

TABLE IV. SUMMARY OF THE FAILURE CONDITIONS OF THE GEOPOLYMER SPECIMENS AT ROOM TEMPERATURE

Mix proportion	0:100	20:80	30:70	40:60	50:50
Failure pattern					
Failure strength (MPa)*	61.37±0.08	78.59±0.09	81.26±0.13	91.44±0.06	96.62±0.15
Elastic Modulus (GPa)*	63.01±0.15	67.05±0.21	69.32±0.32	70.69±0.17	73.16±0.27

*average values calculated with 95% confidence

2) Effect of curing temperature

Fig. 6 shows the relationship between compressive strength and curing temperature of specimens with different mixed proportions. Due to poor rate of geopolymerization at lower curing temperature, most of the geopolymer specimens with different composition ratios gain with the curing temperature up to their optimum curing temperature. And optimum curing temperature of geopolymer with all tested ratio for higher strength lies between 50 °C and 70 °C. Therefore, we can conclude that the optimum curing temperature can be considered as close to 60°C. Geopolymer gains strength when curing temperature is increased because Si and Al existing in the source material is being easily dissolved from the source material with the curing temperature. However, it is notable that the inter granular structure of geopolymer would go through a gradually breaking up process beyond the optimum curing temperature, indicating a possibility of a strength reduction.

B. The variation of elastic modulus with different curing temperature

The variation of elastic modulus of geopolymer with different curing temperature is shown in Fig. 7. Elastic moduli are calculated by measuring the slopes of the uniform elastic regions on the stress strain curves. Elastic moduli (E) for all tested sample are in the range of 6 - 11 GPa, and it presents a similar tendency with compressive strength.

Thus, the stiffness of the geopolymer increase with the curing temperature before optimum curing temperature (60 °C) and then undergo a reduction with temperature.

C. Comparison with other Geopolymers

Next, the mechanical strength of different geopolymers were studied (refer Fig. 8). In Fig. 8, class G cement (GC - currently used in the petroleum industry) was obtained from previous work by Nasvi et al. [17]. Further, the geopolymer specimens prepared using ASTM class F Fly ash based (FA) on the mix design was considered [17]. The composition of the G cement and the fly ash used for comparison are listed in Table V. Even made from different raw materials, the density of geopolymer, ratio of alkaline liquid/binder, as well as Na₂SiO₃/NaOH proportion were all at the same value. For the comparison, 50:50 fired siltstone and slag geopolymer samples were used.

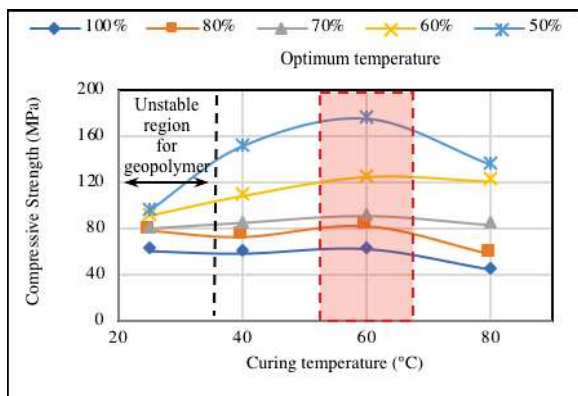


Fig. 6. Variation of mechanical strength with curing temperature

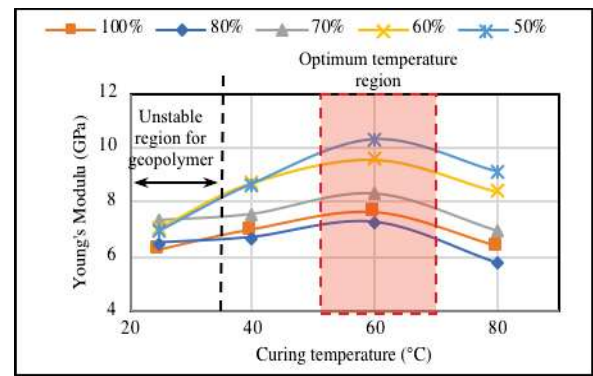


Fig. 7. The variation of Young's modulus and curing temperature

Comparatively, the three materials share the same tendency of variation for UCS values where the optimum temperature is around 60 °C (Fig. 8(a)). Further, the fired siltstone-slag based geopolymer (FS) possesses better performance on mechanical strength than the others (Fig. 8(a)). The peak strength that FS could bear is 176.17 MPa, which is significantly larger than the compressive strength FA and GC, which are 88 MPa (around 50% lesser) and 52 MPa (around 70% lesser), respectively [17]. As the curing temperature increases, the reduction rate of FA is less than that of FS, but its failure strength is still significantly less than the latter. Interestingly, at lower temperatures, GC performs well than FA while FS has the highest strength among all.

Similarly, for E values, FS, GC and FA exhibits a similar trend at lower and higher temperature ranges. However, the optimum temperature shows a considerable variation. For example, FS has the highest E and strength values at the same optimum temperature of 60 °C. The optimum values have been increased by 10 °C for FA while it has been reduced by 20 °C for GC (refer Fig. 8(b)). This might be due to the different Si/Al ratios of the specimens. For example, FA has a higher Si/Al ratio (refer Table V) and in comparison, FS and GC has a lower Si/Al ratio (refer to Table II and III) causing lower stiffness at elevated temperatures compared to FA. Nevertheless, FS has the highest E values from lower to higher temperatures.

Hence, FS performs better than the GC or FA for all the temperatures and has the best performance at elevated temperatures (50 – 70 °C). For lower temperatures, GC has higher strength and stiffness than FA and for higher temperatures, its vice versa. Therefore, FA is suitable to use as a well cement at hot regions where the temperature ranges from 50 – 70 °C while GC is suitable to use at reservoirs at cold

TABLE V. COMPOSITION OF G CEMENT AND FLY ASH [18]

Component	Typical values (%)		Component	Typical values (%)	
	G cement	Fly ash		G cement	Fly ash
SiO ₂	21.7	48.3	MgO	4.3	0.4
Al ₂ O ₃	3.2	30.5	Na ₂ O	-	0.2
Fe ₂ O ₃	3.7	2.8	SO ₃	0.74	0.3
K ₂ O	-	12.1	LOI		1.7

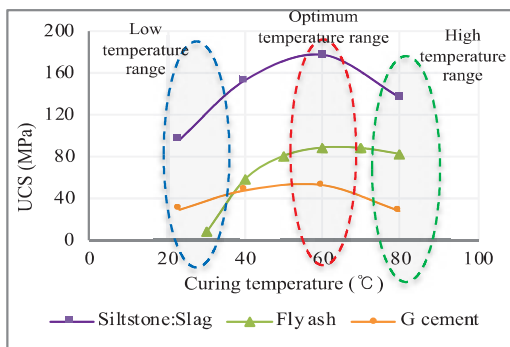
regions (<40 °C), while FS can be used in both regions. However, other influencing factors also need to be tested to confirm the appropriateness for a well cement material, such as durability, permeability and porosity variations and chemical reactions when exposed to CO₂ and adaptive to pressure.

IV. CONCLUSIONS

Based on the finding from this experimental work, the following conclusions are drawn:

- The additional slag added into raw material help geopolymer gain better mechanical strength and stiffness.
- The optimum curing temperature for strength and stiffness of fired siltstone-slag based geopolymer lies in the range of 50 -70 °C.
- The siltstone-slag based geopolymer showed the highest strength and stiffness compared to GC and FA for all temperatures. GC performs well at lower temperatures and FA exhibits higher values at higher temperatures. Therefore, GC would be a better performer at lower depths (<1 km) and FA is for higher depths (>1 km). However, FS is suitable for both lower and higher depths in terms of the effect of downhole temperatures.

(a) UCS variation



(b) E variation

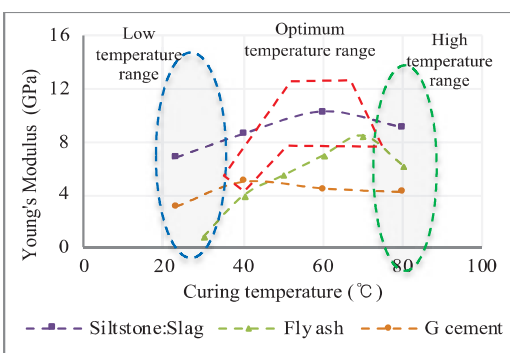


Fig. 8. Comparison of the strength and Young's modulus variation of fired-siltstone based geopolymer, fly ash based geopolymer [17] and G cement [17]

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