Investigating Amorphous Composition Mix Design Performance and Properties of Fly Ash-Based Geopolymer

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ABSTRACT: Identifying viable replacement for Ordinary Portland Cement (OPC) is part of the effort to limit CO_2 emission during its production. Fly ash-based geopolymer composites have emerged as a sustainable and promising replacement product with proven comparable binding properties, coupled with superior characteristic in some area, when compared to OPC. However, fly ash being a by-product of coal combustion, remains unfavourable in industry application due to its deferring composition dependent on the type of coal feedstock, resulted in inconsistent mechanical and chemical properties produced. In order to overcome this shortcoming, a unified method to evaluate fly ash at the amorphous composition level is required. This study aims to tackle this challenge by establishing a framework to effectively identify relationship among the fly ash amorphous content and identifying an optimum proportion of alkaline activator in order to optimise its full potential in geopolymer concrete or mortar production. This is achieved by utilising XRF and Q-XRD analyses on geopolymer specimen's amorphous compositions and evaluate their respective compressive strength through laboratory testing. A number of testing variants are carried out including curing regime, alkaline activator concentrations, presence of mechanical activation and deferring water content are investigated.

KEYWORDS: *geopolymer, fly ash, compressive strength, mortar, mix design*

1 INTRODUCTION

Fly ash-based geopolymer composites have emerged as a sustainable and promising alternative product that can replace OPC with proven comparable binding properties. Fly ash is an industrial waste by-product that has the binding potential, similar to OPC, which can be triggered through the use of alkaline solutions such as combination of sodium hydroxide and sodium silicate to produce geopolymer composites. Fly ash-based geopolymer composite has the potential to be used as construction material due to its high compressive strength, high durability, low shrinkage, acid resistance, fire resistance and low thermal conductivity properties, making it the ideal construction material, without the burden of environmental effect as with OPC.

Geopolymer are synthesized by alkaline and/or silicate activation of a solid aluminosilicate source, resulting in a highly cross-linked amorphous gel binder (Provis et al. 2009). This amorphous gel binder has comparable properties with OPC based binder, making it an ideal alternative cementitious material to form concrete or mortar. Geopolymerization, the process by which geopolymer is synthesized, involves a chemical reaction between the alominosilicate oxides with silicates under highly alkaline conditions. This process yields a three-dimensional polymeric chain and ring structure consists of Si-O-Al-O bonds that makes the binder gel (Xu & van Deventer, 2000). Si-O-Al bridges form subsequently which help the geopolymer to achieve its strength. For fly ash sources with minimum calcium content, which are typically classified as Class F fly ash in accordance to ASTM Standard Specification C 618, the primary type of bonding is N-A-S-H, i.e. sodium aluminium silicate hydrates.

Ever since the introduction of fly ash based geopolymer composite was highlighted as a viable alternative product to replace OPC, its application remains limited in the industry due to a number of reasons. The mechanical properties of geopolymer concrete is highly dependent on the chemical composition of the fly ash used. Fly ash being a by-product produced from coal combustion, has widely differing chemical compositions from one power stations to the other. Due to this indifference in the fly ash amorphous composition despite its readily abundance supply, different source of fly ash cannot be universally adopted in geopolymer manufacturing to produce the intended mechanical and chemical properties consistently.

It is therefore vital to understand the factors affecting geopolymer properties. Past research by Skvara et al. (2005) concluded that properties of geopolymer can be varied by altering the Si/Al, Na/Al ratios and water content. Additional research by William &

van Riessen (2010) also revealed that formulation of a geopolymer mixture using the amorphous composition yielded stronger geopolymer pastes when compared to using the bulk composition, by keeping the atomic ratio Si/Al, Na/Al and H/Si constant. This is because the bulk composition method does not adjust the composition based on the amorphous content, which is responsible for the chemical reaction that resulted in the difference in compressive strength. Therefore, it is believed that optimum fly ash-based geopolymer properties can be replicated by considering its amorphous content for the mixture design and adopting an optimum set of atomic ratios respectively.

This research aims to investigate the influence of specific atomic ratios from two different fly ash sources on the compressive strength of geopolymer. Compressive strength is selected as the main parameter as it is the key performance criterion that needs to be satisfied for structural used. Test samples were prepared using amorphous composition method as highlighted to yield superior compressive strength compare to the well accepted bulk composition method. This research further explored changes in alkaline activator proportion to investigate its impact on compressive strength for each of the fly ash specimens. Other factors that affect the mechanical properties are explored including the effect of mechanical activation and curing condition.

2 MATERIALS & METHODS

2.1 *Fly Ash*

Class F Fly ash has favorable binding properties and chemical and thermal resistant application, when compared to other fly ash type. Two types of fly ash, each in 20 litre containers, were obtained from Gladstone and Eraring Power Stations in Queensland and New South Wales, respectively.

2.2 *Aggregates*

The aggregates used throughout all design mixes consist of fine building sand were obtained from only one source. Sand that was used to prepare the specimens were taken from the same pit in Curtin University's concrete laboratory to ensure consistency. Before use, these aggregates were prepared in accordance to AS 1141.6.2 (1996) to ensure constant moisture content in the fine aggregates was achieved throughout the research.

2.3 *Alkaline Activator Solution*

Alkaline activator solution is required to react with a solid aluminosilicate source, which is found in fly ash, to form geopolymer composites. The proportion of the alkaline activator used differs in each mixture proportion depending on the amorphous content of the fly ash source. The sodium hydroxide pellets used has a purity of 98% that was later watered down to produce the sodium hydroxide solution. The alkaline activator solutions were prepared by mixing sodium hydroxide and sodium silicate solutions (D grade), which has a SiO_2 to Na_2O ratio by mass of 2.0. The chemical composition of sodium silicate of this grade consists of SiO2 = 29.40%, Na2O = 14.70% and H2O = 55.90% by mass. The combined solution was let cool at ambient temperature before use.

2.4 *Mixture Design*

In order to achieve an optimum compressive strength result, the mortar mixture was prepared by adopting the amorphous composition method proposed by Williams & van Riessen (2010), where a mixture design with the required constituent material proportion can be calculate by solving equation (1), as:

$$W = F^{-1}R \quad (1)$$

where $W = [F1\ F2\ F3\ F4]^T$ which is the weight proportion of each mortar constituent in which $F1$ = fly ash, $F2$ = sodium silicate, $F3$ = sodium hydroxide and $F4$ = water. Matrix F is a 4 x 4 matrix, each coefficient is calculated from the number of mole per grams of each controlled element. Matrix R is a column matrix containing the elemetal ratio in term of a fraction of Aluminum, Al, that is, $R = [\text{Si/Al}\ 1\ \text{Na/Al}\ (\text{Si/Al})(\text{H/Si})]^T$.

2.5 *Laboratory Procedure*

The following outlines the sequence for the preparation of fly ash based geopolymer mix specimens:

1. Mixture specimens are prepared with two batches with two different particle sizes. Mechanical activation, i.e. grinding, is applied on the fly ash in one batch with finer particle size in order to achieve high compressive strength. The other fly ash batch without grinding was used as benchmark to prove this hypothesis. Both Gladstone and Eraring fly ash samples were ground for 20 minutes with 50% solid (fly ash) and 50% water. After ground, the fly ash was left idle to allow all water to evaporate.

2. Once the fly ash was ground, the specimens were put through a Laser Particle Size Analysis to obtain its particle size distribution. The analysis was carried out at ALS Ammtec facility.
3. The bulk composition showing each elemental proportion of both fly ash sources was subsequently measured using XRF analysis at ALS Ammtec facility.
4. Crystalline phasing of the fly ash sample was determined through XRD testing. The XRD result can be quantified through Rietveld refinement method and subsequently the crystalline composition was determined using Topas software. This is also known as the Quantitative XRD (Q-XRD) analysis. The analysis was carried out in John de Laeter Centre in Curtin University.
5. Amorphous composition content can now be deduced by subtracting the csrystalline composition content from the bulk composition content, as a fraction of the equivalent oxide. A mixture proportion was deduced from a matrix formulation (refer to section 2.4 Mixture Design) with the weight proportion of fine aggregates, sodium hydroxide, sodium silicate and fly ash established based on the selected atomic ratios of Si/Al, Na/Al and H2O/Si. Their respective range being 1.8 – 2.2, 0.9 – 1.2 and 5.5 respectively in order to achieve the highest compressive strength.
6. Once the constituents of the mortar have been prepared and ready to mix, the mixing phase commenced in accordance to AS 1012.2 (2014). After the geopolymer mortar had been sufficiently mixed, casting took place in accordance with AS 1012.8.1 (2014).
7. Following the casting, the samples were placed to rest in storage cabinet at controlled temperature and humidity at 23°C and 50% relative humidity. After 48 hours, once the moulds were observed to set well enough, they were demoulded and placed in at humidity cabinet under conditions of 38°C and 95% relative humidity for 7 days.
8. Testing commenced in accordance with AS 1012.9 (2014) once the mortar sample had cured for 7 days.
9. After 2 weeks the fly ash based geopolymer mortar samples were observed visually for the extent of the efflorescence present.

3 RESULT

3.1 *Laser Sizing*

Once the fly ash is ground it is necessary to determine the sizing of fly ash particles that are to be used in the experiment. As per Table 1, the particle sizes, for 80% passing, for both Eraring and Gladstone fly ash samples are significantly reduced through mechanical activation. After grinding, particles size is brought down to below 45 μm, which is the recommended threshold by Komljenovic et al. (2010) and van Riessen & Chen-Tan (2013) to achieve larger compressive strength.

Table 1. Percentage Reduction due to Mechanical Activation of Eraring and Gladstone Fly Ash.

Fly Ash	Unground Particle Size	Ground Particle Size	Percentage Reduction
Eraring	57.05 μm	42.64 μm	25.25%
Gladstone	46.60 μm	36.67 μm	21.31%

3.2 *XRF Result*

The elemental oxide composition of Eraring and Gladstone fly ash is provided in Table 2. The result confirmed that both these fly ash sources meet the Class F classification with low CaO content (<10%) in accordance to ASTM 618. The LOI at 1000^0C provides a reasonable estimate of the carbon content present in each fly ash source. The result showed low carbon content and therefore not expected to significantly affect the compressive strength of the mortar.

Table 2. Elemental Composition of Eraring and Gladstone Fly Ash Expressed in Oxide Form.

Element	Eraring Weight %	Gladstone Weight %
Al_2O_3	24.60	26.10
BaO	0.06	0.17
CaO	2.30	3.52
Fe_2O_3	2.24	11.58
K_2O	2.08	0.68
MgO	0.57	1.23
MnO	0.06	0.15
Na_2O	0.76	0.67
P_2O_5	0.08	0.65
SO_3	0.06	0.09
SiO_2	63.80	48.20
SrO	0.06	0.21
TiO_2	0.96	1.41
LOI at 1000^0C	2.07	0.35

3.3 *Q-XRD Result*

The Q-XRD analysis was used to find out the crystalline content of both Eraring and Gladstone fly ash. Table 3 outlines the phase abundance of the crystalline content present in both these fly ash sources.

Table 3. Phase Abundance of Eraring and Gladstone Fly Ash as Determined by Q-XRD.

	Eraring	Gladstone
Component	% Weight in Original Sample	
Quartz	8.116	5.613
Mullite	15.384	10.008
Hermatite	-	2.948
Magnetite	-	5.461
Corundum	0	0

3.4 *Amorphous Phase*

Table 4 outlines the reactive content in both the Eraring and Gladstone fly ash samples. The amorphous content is calculated by subtracting crystalline composition (by Q-XRD) from the bulk composition content (by XRF).

Table 4. Elemental Composition of the Amorphous Content in Eraring and Gladstone Fly Ash.

Element	Amorphous Content of Eraring Fly Ash % Weight	Amorphous Content of Gladstone Fly Ash % Weight
Al_2O_3	13.55	18.91
Na_2O	0.76	0.67
SiO_2	52.34	39.76
Total	65.65	59.34

3.5 *Mortar Mixture Proportion*

Past research such as Duxson & Mallicoat et al. (2007) and Duxson et al. (2005) indicated that the Si/Al ratio that yields the highest compressive strength is 1.8. Given the Eraring fly ash a Si/Al ratio beyond the optimum ratio of 1.8. However, the Eraring fly ash sample has a significant Si content. As a result, the Si/Al ratio needs to be increased above the suggested Si/Al ratio of 1.8 in order to avoid the mixture proportions yielding negative values in the design mix. It is thus hypothesized that the Gladstone fly ash based geopolymer mortar will yield high compressive strength when compared to the Eraring mortar specimens. The Si/Al ratio used for both Eraring and Gladstone mix specimens are provided in Table 5. For the purpose of this research, the ratio for (Si/Al)×(H/Si) was made constant at 11 for all mixture specimens in order to evaluate the effect of Si/Al ratio with a constant water content. H is used rather than H_2O to be consistent with using elemental ratios. The ratio for Na/Al has been kept constant as 0.75 for all mixes.

Table 5. Si/Al Ratios Used for Different Mix Design.

Atomic Ratios	Eraring			Gladstone		
	Mix 1	Mix 2	Mix 3	Mix 1	Mix 2	Mix 3
Si/Al	3.5	3.75	3.9	2	2.2	2.4

Table 6 and Table 7 show the calculated mixture's mass proportions for both Eraring and Gladstone fly ash based geopolymer. The value for *F*1, *F*2, *F*3 and *F*4 were deduced based on William and van Riessen (2010) amorphous method formulation as described earlier. Since sand was not provided as part of the proportion %, it was kept a constant 60% of the total design mix weight for all cases.

Table 6. Mass Required for Each Constituent in the Mix Design for Eraring Fly Ash.

Constituent		Geopolymer Paste (% Proportion)		
		Mix 1	Mix 2	Mix 3
Fly Ash	(*F*1)	72.96	70.90	69.71
Sodium Silicate Solution	(*F*2)	11.29	20.60	25.93
Sodium Hydroxide Solid	(*F*3)	3.67	1.74	0.64
Water	(*F*4)	12.08	6.76	3.72

Table 7. Mass Required for Each Constituent in the Mix Design for Gladstone Fly Ash.

Constituent		Geopolymer Paste (% Proportion)		
		Mix 1	Mix 2	Mix 3
Fly Ash	(*F*1)	66.58	64.66	62.85
Sodium Silicate Solution	(*F*2)	10.90	20.39	29.35
Sodium Hydroxide Solid	(*F*3)	5.34	3.33	1.43
Water	(*F*4)	17.18	11.62	6.37

3.6 *Compressive Strength*

Compressive strength testing commenced once the mixture specimens had been cured for 7 days as considered adequately set for further handling. This research aims to identify the compressive strength trend based on different elemental ratio, and not to compare the data with past research result, which would otherwise require full 28 days compressive strength.

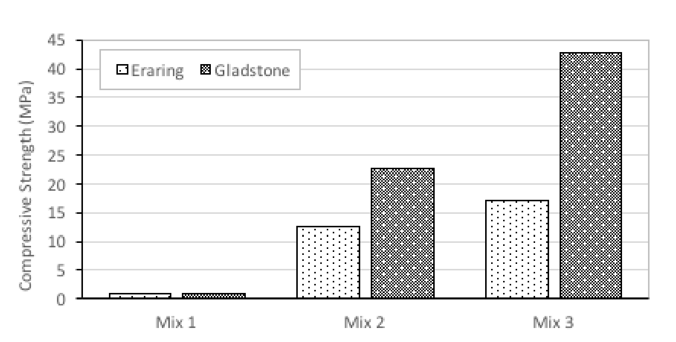

Figure 1. Compressive Strength of Different Design Mixes for Unground Eraring and Gladstone Fly Ash.

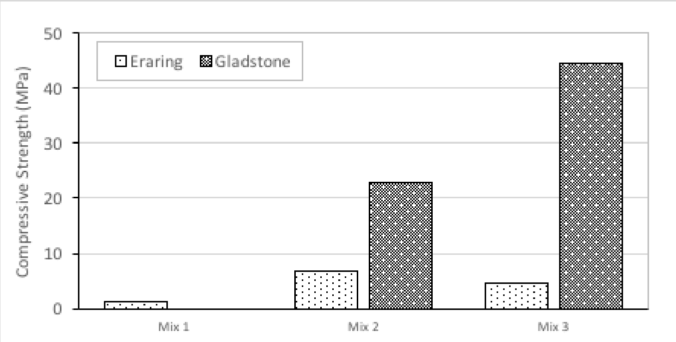

Figure 2. Compressive Strength of Different Design Mixes for Ground Eraring and Gladstone Fly Ash.

It was observed that mixes 2 and 3 for both ground and unground Gladstone fly ash registered higher 7 days compressive strength when compared to the same period in past research findings by Hurst (2011) who used bulk composition mix design. Hence the compressive strength results obtained in this research show that using amorphous composition mix design method can yield higher compressive strength when compared to bulk composition mix design method. This finding is also in line with the same conclusion deduced by Williams and van Riessen (2010).

Refer to

Figure 1 and Figure 2, for both ground and unground fly ash sample, Gladstone fly ash is found to have significant larger compressive strength when compared to Eraring fly ash. This observation aligned well within the expectation where Gladstone fly ash atomic ratio Si/Al is much closer to the optimum ratio of 1.8 compared to Eraring fly ash.

Both Eraring and Gladstone fly ash sample mix 2 contain more sodium hydroxide solution. Despite the expectation that with higher sodium hydroxide solutions, more OH^- would present. This in turn should result in higher compressive strength compared to mix 3 but the result showed otherwise with mix 3 showing higher compressive strength compared to mix 2.

It is worth noting that mix 3 contained a larger amount of sodium silicate solution, which resulted in high Si content. Geopolymer with high Si content is known to be able to create a larger polymer structural network at atomic level based on a research by Rowles & O'Connor (2003), hence resulted in higher compressive strength.

3.7 *Effect of Alkaline Activator Concentration*

Table 8 outlined the proportion of sodium silicate solution and sodium hydroxide solution to achieve the proportion of alkaline activator proportions among each mix. It is evident that for both Eraring and Gladstone fly ash based mortar, an increase in sodium silicate solution is adopted from mix 1 to mix 3, resulted in an increase in Si/Al atomic ratio, which in turn leading to increase in compressive strength.

Table 8. Proportion of Alkaline Activator in Each Design Mix.

	Eraring			Gladstone		
	Sodium Silicate Solution (%)	Sodium Hydroxide Solution (%)	Total % of Design Mix	Sodium Silicate Solution (%)	Sodium Hydroxide Solution (%)	Total % of Design Mix
Mix 1	4.52	6.30	10.82	4.36	4.88	9.24
Mix 2	8.24	3.40	11.64	8.16	5.97	14.13
Mix 3	10.37	1.74	12.11	11.72	3.12	13.84

3.8 *Effect of Water Content*

Water is released during geopolymerisation of fly ash based geopolymer mortar and hence is not part of the chemical reactions. However, water is a crucial component that affects the workability at fresh state and determine the mechanical properties once hardened. Table 9 outlined the additional water added to the mixes to allow the specimens to have adequate workability during casting. The additional water was included in the calculations to determine the water to geopolymer solids ratio by mass, presented in Table 10.

Table 9. Additional Water (mL) Added to the Design Mixes.

	Eraring		Gladstone	
Mix	Unground	Ground	Unground	Ground
1	320	350	200	250
2	275	270	0	0
3	125	290	0	0

Table 10. Water to Geopolymer Solids Ratio by Mass of Each Design Mix.

	Eraring		Gladstone	
Mix	Unground	Ground	Unground	Ground
1	0.40	0.42	0.42	0.45
2	0.38	0.37	0.30	0.30
3	0.29	0.38	0.29	0.29

The water to geopolymer solid ratio by constituent mass is plotted against the compressive strength of the fly ash based geopolymer mortar specimens as presented in Figure 3 and Figure 4 for both Eraring and Gladstone samples. Both graphs clearly show that increase in water to geopolymer solids leads to a decrease in the compressive strength of the geopolymer mortar specimens.

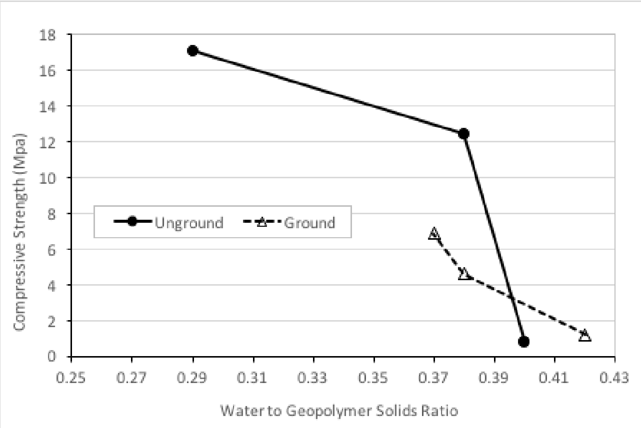

Figure 3. Water-to-Geopolymer Solids Ratio by Mass for Eraring Fly Ash.

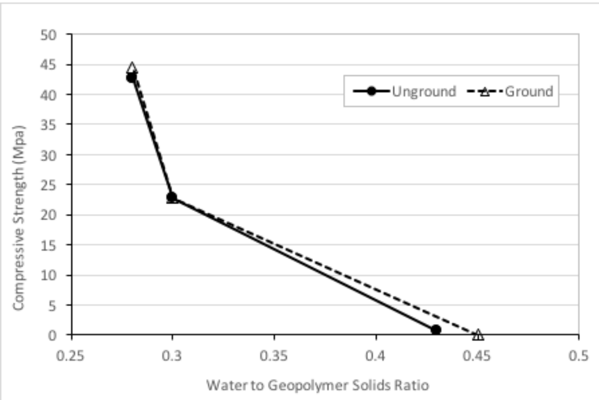

Figure 4. Water-to-Geopolymer Solids Ratio by Mass for Gladstone Fly Ash.

3.9 *Effect of Mechanical Activation*

Figure 6 shows how mechanical activation, i.e., grinding, affected the compressive strength in both for Eraring and Gladstone fly ash respectively. While it was observed that Gladstone samples showed an increased in compressive strength for the ground samples. Eraring samples, on the other hand, had a fall in compressive strength in the ground samples. The reason is believed to be a result of greater water content in the Eraring fly ash mortar sample during mixing. As the mixing activities were carried out on different days with varying ambient temperature and humidity, causing additional drying in the sand in the Eraring fly ash mortar ground mix, hence resulted in a decrease in compressive strength.

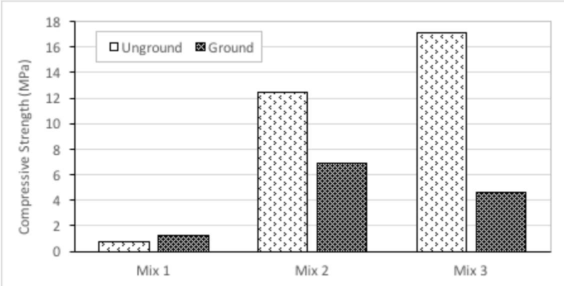

Figure 5. Compressive Strength of Eraring Fly Ash-based Geopolymer Mortars for Unground and Ground Conditions.

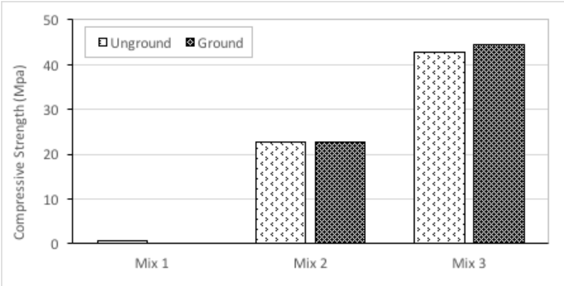

Figure 6. Compressive Strength of Gladstone Fly Ash-based Geopolymer Mortars for Unground and Ground Conditions.

3.10 *Efflorescence Observation*

Figure 7 and Figure 8 show both Eraring and Gladstone fly ash ground unground mortar samples. Comparing all three mixes with the knowledge of their compressive strength, mix 1 is the only sample that does not present visual efflorescence. This is believed to be due to the fact that the alkaline activator solution has fully reacted with the fly ash particles. The efflorescence observed in mixes 2 and 3 was considered minor and therefore it could not be concluded that there is excess of alkaline solutions in these mixes.

Figure 7. Visual Observation of Efflorescence on Unground Eraring Specimens for Each Design Mix.

Figure 8. Visual Observation of Efflorescence on Unground Gladstone Specimens for Each Design Mix.

3.11 *Evaluating Fly Ash Sources*

The vast difference in compressive strengths between Gladstone and Eraring fly ash sources outline the need to evaluate fly ash samples prior to mixing to yield improved mechanical properties. For fly ash samples with high SiO2 content, such as Eraring, another type of alkali compound such as sodium aluminate should be used instead of sodium silicate to reduce the Si/Al ratio. Based on the compressive strength results gained, the maximum compressive strength of the Eraring was not believed to have been achieved. This is due to not having enough OH- ions in the design mix to enable reaction with the excessive Si ion content caused by the high Si/Al ratio.

4 DISCUSSION

4.1 *Amorphous Composition Mix Design Approach*

The benefit of using amorphous mix design approach is its ability to allow various components content to be tailored for the intended optimum functional performance, including compressive strength and workability. This is clear when the amorphous mix design samples from this research are compared against with other samples from past research using the same mix design approach. Table 11 compares the samples contents proportion and their results with past research carried out by William & van Riessen (2010) using the same amorphous composition mix design approach. The values stated in range in this table for this research are based on the result from different mix samples.

4.2 *Effect of Atomic Ratio*

Past researches had revealed that the metakaolin geopolymer formulations optimised for maximum compressive strength was achieved when the nominal composition of Si/Al = 1.8–2.2 and Na/Al = 0.9–1.2 (Rowles et al. 2003, Steveson et al. 2005, and Duxson et al 2007). This optimum range of Si/Al had often been quoted for optimised fly ash application as past research by Fernández & Palomo (2005) showed that the microchemistry of strength optimised fly ash geopolymers have Si/Al and Na/Al ratio similar to that of metakaolin systems.

Using XRF and Q-XRD analysis, it was found that Eraring fly ash has a much larger SiO_2 content than the optimum 1.8 Si/Al ratio. When there is a large Si/Al ratio present, OH^- ions from the alkaline reactivator solutions becomes insufficient to dissolve the Al^{3+} and Si^{4+} ions provided by the fly ash source, which ultimately lead to a decrease in compressive strength. Gladstone fly ash, on the other hand, has a near ideal Si/Al ratio and therefore it is evidenced that relatively higher compressive strength was reached. Past research showed that at optimum Si/Al ratio, an increase in the Si-O-Al bridges can formed resulted in higher number of N-A-S-H bonds which contributed to the compressive strength.

Table 11. Samples Proportion and Results Comparison with William & van Riessen (2010).

Constituent	This Research		William & Van Riessen (2010)	
	Gladstone	Eraring	Collie	Pt Augusta
Fly Ash	62.85–66.58	69.71–72.96	66.1	67.1
Sodium Silicate Solution	10.90–29.35	11.29–25.93	14.9	12.5
Sodium Hydroxide Solid	1.43–5.34	0.64–3.67	5.7	6.0
Water	6.37–17.18	3.72–12.08	13.4	14.4
Si/Al ratio	2-0–2.4	3.5–3.9	1.7	1.7
Compressive Strength (Unground)	0.76–42.75 MPa	0.75–17.09 MPa	29 MPa	48 MPa
Additional Water Added	125–300 mL	0–200 mL	N.A.	N.A.

Table 11 shows that all samples in both researches that have a Si/Al ratio between 1.7–2.4 yielded relatively high compressive strength when compared to the Eraring sample which has a Si/Al ratio of 3.5–3.9 that falls outside the optimum range.

To further improve Eraring fly ash compressive strength, it is suggested that sodium silicate solution to be replaced by sodium aluminate solution in order to reduce the current Si/Al ratio. This research showed that Si/Al ratio of 2.4 was found to have a reduced water-to-geopolymer solids ratio as the sodium silicate reacted directly with the amorphous content of the fly ash to produce N-A-S-H bonds within the geopolymer.

4.3 *Effect of Alkaline Activator Concentration*

The role of sodium hydroxide was not seen to greatly affect the compressive strength in the current research samples. Although past research suggested that high sodium hydroxide content can contribute to final compressive strength, the current research mix design setup was not able to prove this outcome with all three mixes for both fly ash sources' higher compressive strength being dominated by high Si content.
It was observed in current research that alkaline activator solution that has a higher sodium silicate solution proportion led to samples with significant higher compressive strength. This is evidenced in sample that has a relatively lower Si/Al ratio.

4.4 *Effect of Water Content on Workability*

This research found that an increase in the water content in the fly ash based geopolymer mortar lead to a reduction in the compressive strength. This is observed for both the fly ash sources and for both ground and unground specimens. Similar to OPC based mortar, increased water content leads to a reduction in compressive strength but allows increased in workability. This is obvious as shown in Table 11 where compressive strength had been compromised with the additional water added to both Eraring and Gladstone sample in this research on top of the calculated water content by amorphous mix method in order to achieve higher workability.

4.5 *Effect of Mechanical Activation*

This research verified past research findings showing that mechanical activation, for a reduction in the particle size below 45 μm, can result to increase in the mortar compressive strength. This is observed this is observed in the Gladstone fly ash ground samples compared to the unground samples. The increase in fly ash surface particles surface area after grinding provides greater reaction with the alkaline activator, ultimately resulted in more complex structural bonds formed chemically that leads to higher compressive strength. On the other hand, the Eraring fly ash ground samples observed a decrease in the compressive strengths when compared to the unground samples. Even though water content for both ground and unground samples was the same, it is believed that sand used in the ground samples mix may have a lower moisture content during the day of mixing with higher ambient temperature and lower relative humidity, and therefore absorbed more water within the mix.

4.6 *Potential of Efflorescence*

Efflorescence is one of the key limitations of geopolymer composite where the term is referring to the formation of white deposits on the geopolymer concrete or mortar. These white deposits are formed when excess alkaline activator remains unreacted with the fly ash particles. It is an indication that alkaline solutions used in the mix has been overdosed. Formation of efflorescence tends to be larger in a humid environment as opposed to a dry environment. Past research by Skvara et al. (2009) found that Na bond in the aluminosilicate binder structure is weaker in a humid curing conditions and thus can become separated from the binding material, especially when the alkaline activator solution is overdosed, leading to formation of efflorescence.

For both the Eraring and Gladstone fly ash based mortar, mix 1 contains no visual efflorescence. This indicated that the alkaline solution has fully reacted with the fly ash particles, but due to the low compressive strength exhibited, it is suggested that there is inadequate alkaline activator used for this mix to adequately form N-A-S-H bonds. On the other hand, both mix 2 and 3 exhibited minor visual efflorescence and showing relatively high compressive strength. However, this research is unable to conclude that mix 2 and 3 has excessive alkaline activator solutions due to only minor efflorescence observed. The higher alkaline activator solution content could not be attributed to the increase in compressive strength either, which the later was dominated by the higher Si/Al ratio.

4.7 *Effect of Curing Conditions*

This research found that fly ash based geopolymer mortar compressive strength gain relies heavily on a controlled curing condition, where elevated temperature and humidity is closely monitored. Table 11 showed that William & van Riessen (2010) research samples had a relatively higher compressive strength than those in this research may be due to the fact that they were cured at a higher temperature of 75°C for 24 h in an electric oven. This remains as a limitation for its wide application in real life industry application as such curing condition cannot be practically and readily made available. Despite the fact that amorphous mix design approach can contribute to a higher compressive strength when compared to the conventional bulk mix design approach, without the curing condition, there is no certainty that the intended compressive strength can be achieved.

5 CONCLUSIONS

The current research supports the use of fly ash based geopolymer composite as a viable alternative to OPC based concrete or mortar. The following outline critical consideration for fly ash based geopolymer composite application and recommendation on its improvement.

1. Larger Si/Al ratio beyond 1.8 can achieve higher compressive strength despite being stated as optimum in past research. This research identified ideal range for Si/Al being no higher than 2.4.
2. The formulation for fly ash-based geopolymer composite using the amorphous composition method produced higher compressive strength compared to using the bulk composition method.
3. When encountered with fly ash source that is high in SiO_2 content, such as the Eraring fly ash, the Si/Al ratio may go beyond the optimum range, resulted in lower compressive strength. To address this unfavourabel outcome, this research recommends to replace sodium silicate solution with sodium aluminate solution in order to adjust the Si/Al ratio back to the optimum range.
4. Fly ash based geopolymer composite compressive strength can be further increased by controling the Si/Al content through the amount of the alkali solution in the alkailine activator mix.
5. Conventional method for fly ash based geopolymer mix design using H_2O/Si ratio should be minimised as excessive water can compromise compressive strength. Instead, alkaline solutions can be added prior to water, which the latter is only added if necessary for the desired workability.
6. Mechanical activation by grinding can be applied to all fly ash sources to improve its composite compressive strength.

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