

PROPOSED SIMPLIFIED METHOD OF GEOPOLYMER CONCRETE MIX DESIGN



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The research aims to determine the best combination of the controlling factors that govern geopolymer concrete's mechanical and physical properties by utilizing industrial waste. Therefore, a review on the controlling factors was conducted. Firstly, it is to identify the controlling factors, namely chemical composition, alkali activation solution, water content, and curing condition. Secondly, understanding the relationship between these controlling factors and the properties of geopolymer concrete. These factors are analysed to the mix proportion components. Finally, a new proportion method is proposed based on combining ACI 211 standard and recommended molar ratios of oxides involved in geopolymer synthesis. The effect of aggregate has been taken into account by applying the absolute volume method in mix design. Based on the results of the study, it is expected to determine the optimal mix proportions based on multi-responses.

Keywords: geopolymer concrete, industrial waste, controlling factors

1. INTRODUCTION

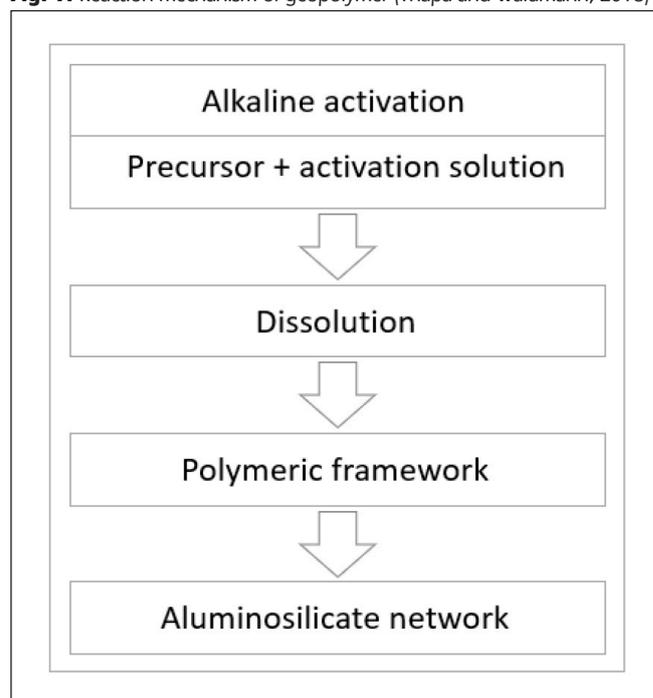
From an environmental point of view, the carbon-dioxide (CO₂) emission has been being increased tremendously due to energy consumption, transportation, and industry. Even though cement plays a vital role in infrastructure construction, it involves an immense emission of carbon dioxide. Statistics showed that 1 ton production of cement produces about 1 ton of CO₂. Therefore, geopolymers are used as an alternative way to reduce the emission of carbon-dioxide caused by cement processing (Davidovits, 1991; Davidovits, 1993). The patent of the geopolymer chemistry concept was introduced by Geopolymer Institute in 1979. This patent was the key to develop new binder materials. Consequently, the high-strength geopolymer cement was invented by Joseph Davidovits and James Sawyer in 1983 (Davidovits, 2002). The source of geopolymer binders can be either natural or synthetic aluminosilicate. The idea of geopolymerization is that the chemical reaction between aluminosilicate oxides and alkali polysilicates produces polymeric (Si-O-Al) bonds of amorphous to semi-crystalline three-dimensional silico-aluminate structures (Davidovits, 1991). Interestingly, it is found that most waste materials are sources of silica and alumina. As a result, these waste materials could be operated in geopolymerization reaction and being binder materials (Van Jaarsveld, Van Deventer and Lorenzen, 1998). In the case of natural sources used to produce geopolymers such as clay, high temperature is needed to calcine the clay, which is about 600 °C (Mlinárik and Kopecskó, 2013). On the contrary, geopolymer binders using waste materials are already calcined from other processes, so they do not need to be calcined (Merabtene et al., 2019; Tchakoute Kouamo et al., 2012). Therefore, utilising waste materials in the construction industry will improve both the sustainability and economics of infrastructure systems (Van Jaarsveld et al., 1998). The reaction

mechanism of geopolymer can be shown in Fig. 1 (Thapa and Waldmann, 2018).

2. TYPES OF INDUSTRIAL WASTE MATERIALS USED AS GEOPOLYMER BINDERS

Industrial waste based geopolymers do not have a unique chemical structure. Their properties are most dependent on their base material characteristics, namely: chemical compo-

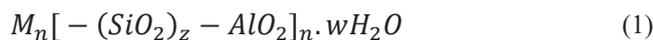
Fig. 1: Reaction mechanism of geopolymer (Thapa and Waldmann, 2018)



sition, the content of glassy phase, amount of soluble silicon and aluminium, particle size distribution, and presence of inert particles (Zhang et al., 2018). Therefore, it is crucial to identify the sources of base materials of geopolymer synthesis, which are under conducting a study. There are many types of waste and by-product materials that have been utilised in geopolymer synthesis, for example, fly ash, blast furnace slag, bottom ash, red mud, rice husk ash, bottom ash, palm oil fuel ash, waste paper sludge ash, tailing metals, silica waste, ceramic waste, etc., (Abdel-Ghani et al., 2018; Bakharev et al., 1999; Chawakitchareon, 2013; Chi, 2016; Ekaputri, Baihaqi and Aji, 2011; Ekaputri, Junaedi and Bayuaji, 2015; Erdogan, 2015; Hanjitsuwan et al., 2017; Junak et al., 2014; Karakoç et al., 2014; Karrech et al., 2019; Khater and Abd El Gawaad, 2016; Kim et al., 2014; Kopecký et al., 2019; Kovalchuk et al., 2007; Malkawi et al., 2016; Mucsi et al., 2020; Musaddiq Laskar and Talukdar, 2017; Monita et al., 2016; Ridzuan et al., 2014; Saeli et al., 2019; Sindhunata et al., 2006; Shoaie et al., 2019; Yankwa Djobo et al., 2016; Ye et al., 2016).

3. CHEMICAL COMPOSITION AND SYNTHESIS

The geopolymer reaction is achieved by the reaction of aluminate-silicate with the availability of alkali activator at low temperature. The following general formula below describes the chemical composition (1):



where M is an alkali cation; z is an integer; n is the degree of polymerisation and w is the molar amount of water (Davidovits, 2002). *Table 1* shows an example of the chemical composition of slag and fly ash conducted using X-ray fluorescence (XRF) (Li et al., 2018). The chemistry matrix is a function of four variables, namely: Si/Al ratio, alkali activator type and concentration, curing temperature, and water content (Duxson et al., 2005; Duxson et al., 2007b).

3.1 Influence of Si/Al ratio

The basic structure of geopolymers is the network structure of (SiO_4) tetrahedrons and (AlO_4) tetrahedrons, which are connected by mutual oxygen atoms. Si/Al ratio reflexes this structure and plays a vital role in geopolymer behaviour. The contribution of the Si/Al ratio comes from the base material of geopolymer. Although the Si-O-Si bonds are stronger than the Al-O-Si bonds, the geopolymer's high performance occurs at an intermediate Si/Al ratio at a certain range of alkalinity. This optimum Si/Al ratio differs for different base material geopolymers, and it is also dependent on processing conditions. It is found that some silicate would not participate in reactivity, such as silicate in quartz. In other words, the amorphous component is the reactive compound. Fur-

thermore, even some amorphous silicate could be prevented from being reacted (Ahmari, Zhang and Zhang 2012; Duxson et al., 2007a; Williams and Van Riessen, 2010; Zheng, Wang and Shi, 2010). Geopolymers with ground granulated blast furnace, GGBS, exhibit better performance at a low Si/Al ratio compared to low-calcium geopolymers (Kubba et al., 2018). The Si/Al ratio can be controlled by adding small silica fume content (Kovalchuk et al., 2007). *Table 2* shows different optimum Si/Al ratios for different materials and the corresponding compressive strengths.

Table 2: Optimum Si/Al ratios for different geopolymers and their compressive strengths

Material	Si/Al ratio	Curing mode	Comp-res-sive strength (MPa)
MK ¹ (Duxson, Mallicoat, et al. 2007)	1.9	Heat curing	78
RM ² : FA ³ (Zhang, He, and Gambrell 2010)	3.2	Ambient curing	13
RM ² : FA ³ : SF ⁴ (Singh, Aswath, and Ranganath 2018)	5.1	Heat curing	32
RM ² : FA ³ : SF ⁴ (Singh et al. 2018)	4.0	Ambient curing	30

MK = metakaolin; RM = redmud; FA = fly ash; SF = silica fume

3.2 Influence of alkali solution

Generally, hydroxide and silicate-based solutions can be used individually or proportionally mixed to synthesize geopolymers. The type and concentration of alkali solutions (hydroxide, silicate-based, and water) have an important impact on geopolymer performance (Chindaprasirt et al., 2007; Fernández-Jiménez and Palomo, 2003; Hardjito et al., 2004; Risdan-areni and Ekaputri, 2015; Tuyan et al., 2018).

Usually, sodium silicate, Na_2SiO_3 ($Na_2O + SiO_2 + H_2O$), is used as a silicate-based solution and could be proportionally mixed with either sodium hydroxide, NaOH, potassium hydroxide KOH, or both (Hardjito et al., 2004; N. Li et al., 2018). In the case of sodium silicate, the activator variables are defined by the silica modulus (M_s) or Na_2O content. Silica modulus is measured in the molar ratio of SiO_2/Na_2O . Na_2O content is calculated as a percentage of the weight of raw material in dry condition (Silva et al., 2019). Increasing these variables for a particular value will decrease the porosity of mixtures. Accordingly, the density would be improved and producing maximum compressive strength values (Tuyan, Andiç-Çakir and Ramyar, 2018). Hydroxyl ions could be measured by molarity. The optimum concentration of NaOH is dependent on curing temperature. When the curing temperature is increased, the required optimum concentration of

Table 1: Chemical compositions of slag and fly ash (wt. %). LOI is the loss of ignition

Material	Oxide								
	SiO ₂	Al ₂ O ₃	CaO	MgO	K ₂ O	Fe ₂ O ₃	Na ₂ O	SO ₃	LOI
Slag	33.81	14.78	38.81	7.09	0.44	0.36	0.26	2.49	1.40
Fly Ash	54.22	31.18	1.24	0.47	1.34	2.36	0.49	0.35	3.25

NaOH increases (Ahmari et al., 2012).

In the case of geopolymers that have GGBS, NaOH concentration plays a vital role in altering the geopolymerization process and affecting the mechanical and physical properties. When NaOH concentration is low in alkali solution, calcium will be dissolved, contributing to the formation of CSH gel. This process yields homogenous and dense products because CSH works as a micro-aggregate. On the other hand, a high dosage of NaOH will be responsible for calcium hydroxide formation, which will prevent the formation of CSH gel. In this case, the variable parameters will be (low-calcium raw material / high-calcium raw material) by weight and $\text{Na}_2\text{O}/\text{SiO}_2$ in the molar ratio (Kubba et al., 2018; Yip et al., 2005).

It should be noted that the unburnt carbon behaves as an inert particulate, which can increase the demand for activation solution due to absorption (Gunasekara et al., 2015). Recently, some geopolymers have been investigated with respect to mechanical activation as a partial and full replacement of chemical activation. They showed good response and developed high compressive strength values when used with activators (Y. Li et al., 2019)

3.3 Influence of curing mode

The curing temperature has a significant influence on optimising geopolymer properties because of related water evaporation. However, a very high curing temperature could be harmful and destabilise geopolymerization (Shoaei et al., 2019). In general, the heat-curing regime is mostly adopted in geopolymer applications. The heat-curing regime is expressed by two components. The first component is curing time, which is ranged from 4 hours to 96 hours with an optimum practical value of 24 hours. The other component is the temperature, which is started from the minimum value of 30 °C up to a maximum temperature of 90 °C.

Curing can be conducted by steam-curing, curing in covered moulds, or dry-curing. The type of curing affects the total porosity, the average pore diameter, and microstructural characteristics (Assi et al., 2016; Hardjito and Rangan, 2005; Jaydeep and Chakravarthy, 2013; Lloyd and Rangan, 2010; Kovalchuk et al., 2007). Interestingly, GGBS geopolymers can be optimised at a much lower curing temperature than low-calcium geopolymers (Kubba et al., 2018). It should be noted that there are some flexibilities in the heat-curing regime. First of all, the heat-curing can be postponed for up to five days with no degradation (Hardjito and Rangan, 2005). In precast concrete, sometimes it is needed to remove the moulds before the ending of curing time to use them in another casting. Therefore, the two-stage curing is valid. This flexibility is valuable in practical use when required to remove the moulds during the curing time (Hardjito and Rangan, 2005). However, full curing out-side the moulds is still controversial (Assi et al., 2016).

3.4 Influence of water content

The influence of water content is represented by a single parameter of which water-to-geopolymer solids ratio by mass, W/G.S ratio. This parameter has a tremendous effect on the compressive strength and workability of geopolymer concrete. The total water mass is equal to the summation of water in the sodium silicate solution, the water that is used to produce the sodium hydroxide solution, and the extra water, if any is needed, should be taken into account. On the other

hand, the geopolymer solids mass should contain the dry raw materials and the solids of the activator solution, for example, the solids of the sodium hydroxide solution and sodium silicate solution (Na_2O and SiO_2) (Assi et al., 2016; Hardjito & Rangan, 2005). The increase of water-to-geopolymer solids ratio increases the workability of concrete. However, there is an optimum value of water-to-geopolymer solids ratio to achieve the maximum compressive strength at acceptable workability (Shoaei et al. 2019). This optimum value is affected by the type of raw materials and activator type (Assi et al., 2016; Kovalchuk et al., 2007; Shoaei et al., 2019; Shoaei et al., 2019).

4. GEOPOLYMER CONCRETE (GPC)

The main difference between geopolymer concrete (GPC) and conventional Portland cement based concrete is the binder, which in case of geopolymer concrete is including the raw material of geopolymer and the alkaline activator. However, the conventional methods that are used in the production of Portland cement concrete (PCC) can be utilised to produce geopolymer concrete. Fig. 2 shows a typical description of one cubic meter of the volume of Portland cement concrete and geopolymer concrete (Lloyd and Rangan, 2010; N. Li et al., 2019)

5. PROPOSED SIMPLIFIED METHOD OF GEOPOLYMER CONCRETE MIX DESIGN

A simplified mix design is proposed by combining ACI 211 (2009) standard and recommended molar ratios of oxides involved in geopolymer synthesis, where

- (i) the desired compressive strength is targeted, and
- (ii) the workability would be verified for the acceptable range based on absolute volume according to the standard (ACI 211, 2009).

The mix design is based on the similarity between the Portland cement concrete and geopolymer concrete mixtures and takes into account different properties of geopolymer concrete.

Fig. 2: Characterisation of Portland cement concrete (PCC) and geopolymer concrete (GPC) in 1 m³ (N. Li et al. 2019)

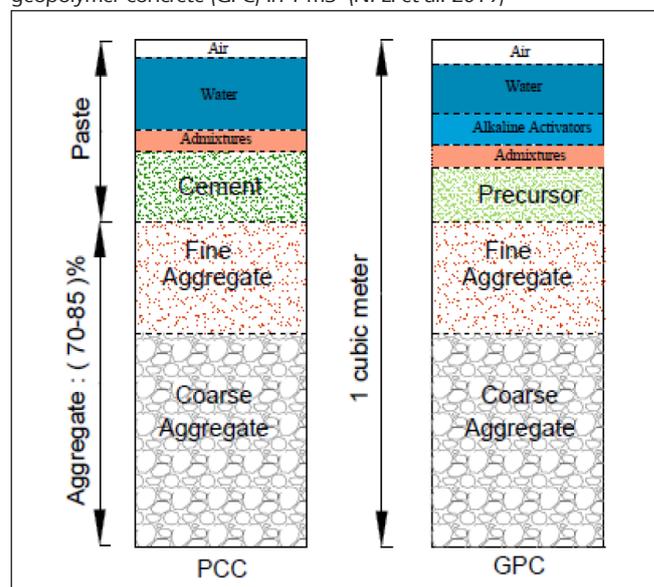


Table 3: Approximate mixing water and air content requirements for different slumps and maximum aggregate sizes for non-air-entrained PCC (ACI 211, 2009)

Slump	Water quantity in kg/m ³ for the nominal maximum aggregate size (mm)							
	9.5	12.5	19	25	37.5	50	75	100
25 – 50	207	199	190	179	166	154	130	113
75 – 100	228	216	205	193	181	169	145	124
150 – 175	243	228	216	202	190	178	160	-
Entrapped air (%)	3.0	2.5	2.0	1.5	1.0	0.5	0.3	0.2

5.1 Water content

According to ACI 211 (2009) standard, the maximum water content can be determined from the maximum size of aggregate, as is shown in *Table 3*.

5.2 Alkaline activator solution content

In case there is no extra water needed to be added to the mix, the water content is only provided from the alkaline activator solution. According to (Heath, Paine and McManus, 2014), the mix oxide molar ratios can be used to produce geopolymers in case of using sodium or potassium hydroxide and silicate (Na₂O.nSiO₂ or K₂O.nSiO₂) activators as illustrated in *Table 4*, where M is Na or K. The alkaline solution will be selected in terms of molarity and concentration according to the chosen water content, see *Table 1* and *Table 4*. If the alkaline solution selection requires less water, the remaining amount of water will be added as extra water to the mixture.

Table 4: Mix oxide molar ratios of alkali activators

Oxide ratio	Molar ratio range
SiO ₂ : Al ₂ O ₃	3.5 – 4.5
*M ₂ O : SiO ₂	0.20 – 0.28
H ₂ O : *M ₂ O	15.0 – 17.5
*M ₂ O : Al ₂ O ₃	0.80 – 1.20

Notation: *M is stands for either Na or K

5.3 Water-to-geopolymer solids ratio

In conventional concrete, the compressive strength at the age of 28 days is considered to determine the water to cement ratio according to ACI 211 (2009) standard. Similarly, the ratio of the water-to-geopolymer solids can be selected from the standard water to cement ratio curve (*Fig. 3, Table 5*) (Pavithra et al., 2016; ACI 211, 2009)

Table 5: Relationship between water-cement ratio and compressive strength of Portland cement concrete, according to ACI 211 (2009) standard

Compressive strength at 28 days (MPa)	Water-cement ratio
41.0	0.41
35.0	0.48
28.0	0.57
21.0	0.68
14.0	0.82

5.4 Raw material content

After determining the water content and water-to-geopolymer solids ratio (W/GS), the geopolymer solids content (GS) can be calculated (2-5):

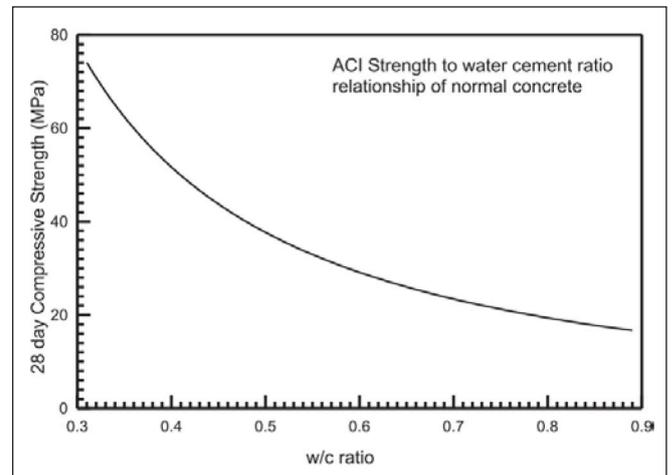


Fig. 3: Strength versus water to cement ratio curve (Pavithra et al., 2016)

$$GS = \frac{W_{content}}{W/GS} \quad (2)$$

$$GS_{SS} = m_{SS} * \% GS_{SS} \quad (3)$$

$$GS_{SH} = m_{SH} * \% GS_{SH} \quad (4)$$

$$GS = GS_B + GS_{SS} + GS_{SH} \quad (5)$$

, where GS is geopolymer solid content; GS_{SS} is solid content of Na₂SiO₃; GS_{SH} solid content of NaOH; m_{SS} is the content of Na₂SiO₃ solution; m_{SH} is content of NaOH solution; GS_B is raw material content.

5.5 Air content volume

The percentage of entrapped air in conventional concrete is illustrated in *Table 3*, depending on the maximum size of aggregate. However, for fly ash-based geopolymer, the air content was found greater than the conventional concrete for the same corresponding size of coarse aggregate based on trial mixes. For the maximum coarse aggregate size of 19 mm, the air content volume percent is assigned two, according to ACI 211 (2009). On the other hand, the air content volume percent of fly ash-based geopolymer was found 3.29 for maximum coarse aggregate size of 20 mm (Ferdous, Kayali and Khenane, 2013). This difference indicates that the entrapped air percent in geopolymer concrete would be greater than it is in conventional concrete. In this proposed method, the entrapped air content in geopolymer concrete will be taken equal to 3.29 V% based on the results of (Ferdous, Kayali and Khenane, 2013).

5.6 Addition of super-plasticiser

In fact, geopolymer concrete is stiffer and stickier than conventional concrete. Therefore, the same amount of water in

geopolymer concrete would produce decrease in workability. Workability can be increased either by increasing the water amount or adding super-plasticiser such as carboxylic ether polymer-based super-plasticiser or naphthalene-based super-plasticiser. Increasing the water amount has a much more negative effect on the strength of geopolymer concrete than adding super-plasticiser. Thus, the super-plasticiser addition is a better choice to increase the workability of geopolymer concrete. The super-plasticiser recommended dosage ranges from 0.8 to 1.5% of binder content (Ferdous et al., 2013; Pavithra et al., 2016; Reddy and Naqash, 2020).

5.7 Coarse aggregate volume

According to ACI 211 (2009) standard, the coarse aggregate volume can be selected depending on two criteria, namely the nominal maximum size of coarse aggregate and fineness modulus of fine aggregate, as is shown in *Table 6*. It should be noted that coarse aggregate volumes are based on oven-dry-rodded weights in accordance with ASTM C29 (ASTM:C29/C29M-09 2009, ACI 211 2009).

5.8 Fine aggregate content

Since all other ingredient volumes are determined, the remaining volume percentage represents the volume percentage of fine aggregate (ACI 211, 2009).

5.9 The moisture content of aggregate

The moisture of aggregate affects two parameters, namely weight of aggregate and content of mixing water. The adjustment of aggregate weight and mixing water content depends on the saturation degree of batched aggregate (ACI 211, 2009).

6. MIXING, CASTING AND COMPACTING OF GEOPOLYMER CONCRETE

One of the most distinctive characteristics of geopolymer concrete is the alkaline activator solution. The most used activator solutions are sodium hydroxide and sodium silicate. The sodium hydroxide solution is prepared by dissolution of sodium hydroxide pellets in distilled water. After that, the solution should be isolated from the atmosphere as much as possible to prevent the possible reaction with atmospheric carbonate for at least 24 hours. Sodium silicate solution can be provided by manufacturers in specific concentrations. Sodium silicate is usually used in combination with sodium hydroxide. In this case, the solution is prepared by dissolution of sodium silicate

Table 6: Coarse aggregate volume in 1 m³ of PCC

Nominal max. aggregate size,mm	Fineness modulus of fine aggregate			
	2.40	2.60	2.80	3.00
9.5	0.50	0.48	0.46	0.44
12.5	0.59	0.57	0.55	0.53
19.0	0.66	0.64	0.62	0.60
25.0	0.71	0.69	0.67	0.65
37.5	0.75	0.73	0.71	0.69
50.0	0.78	0.76	0.74	0.72

in sodium hydroxide to obtain the required concentration. The solution should be prepared at least 24 hours before it is used in mixing to allow the necessary equilibrium (Ahmari et al., 2012; Duxson et al., 2005; Duxson et al. 2007b).

The addition of amorphous silica with sodium hydroxide can replace the use of sodium silicate since the alkali activator is the most expensive component in geopolymer concrete (Heath et al., 2014; Pavithra et al., 2016). After the activator solution is being ready to use, the raw material and aggregate should be mixed dry for at least three minutes. Then the alkaline liquid should be added after it is mixed with the super-plasticiser and the extra water if it is needed just prior to mixing. The wet mixing time should last for four minutes at least. The fresh concrete can be handled and formed up to 120 minutes after mixing (Hardjito and Rangan, 2005). Based on our experience, we were not able to mix geopolymer concrete, when first the dry material (precursor and aggregate) is mixed, then thereafter the alkali activator solution was added, similarly to the mixing method in case of PCC. In case of geopolymer mortar or concrete first the liquid gel (alkali activator solution + precursor + super-plasticiser) formation is achieved, then the aggregate is added and mixed (Kopeckó et al., 2017). It is essential to make trial mix before starting the main experiments.

The compaction of geopolymer concrete is as same as it is in conventional concrete (Hardjito and Rangan, 2005).

7. CONCLUSIONS

The following conclusions and future work can be stated:

- The controlling factors (chemical composition, alkali activation solution, water content, and curing condition) of geopolymer are sensitive to the source material.
- Heat curing limits the use of geopolymer concrete in practical applications. For this reason, the use of geopolymer concrete is primarily limited to the precast concrete application.
- The cost of geopolymer concrete synthesis with sodium silicate is relatively high.
- Herein a new simplified geopolymer concrete (GPC) mix design is proposed based on the Portland cement concrete (PCC) mix design (ACI 211, 2009) with the combination of the recommended molar ratios of oxides involved in geopolymer synthesis. This simplified method will allow us to optimize the controlling factors of geopolymer concrete to produce optimum compressive strength with acceptable workability. This process will be conducted by utilizing the common factors between PCC and GPC, namely water and aggregate.
- In future work, it is essential to investigate the possible replacement of sodium silicate by amorphous silica such as silica fume, rice husk ash, or ground waste glass in the activator solution to reduce the cost of production.

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