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Factors affecting the slump and strength development of geopolymer concrete

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Abstract

Cement production is estimated to be responsible for 5-8% of global total carbon dioxide (CO₂) emissions. Geopolymer concrete (GC) is claimed to release up to 45% less CO₂ for a comparable concrete, but is more difficult to manufacture. This study investigated the effect of factors other than mix design on the slump and strength development of GC produced from low-calcium fly ash (FA) and up to 50% ground granulated blastfurnace slag (GGBS) replacement; these were: curing methods and temperatures (at 10, 20 and 75 °C); FA fineness; superplasticiser type; water content; and GGBS/FA ratio. Methods are also presented for the volume-to-mass conversion of sodium hydroxide at a specific molarity and ambient temperature, and an effective combination of wax-based mixtures as mould agents to overcome the inherent manufacturing mould release difficulties. Steam-curing improved the compressive strength of the FA-based GC over oven drying by up to 20%, as did increasing the FA fineness (although this became negligible with 50% GGBS replacement). Both naphthalene and polycarboxylate superplasticizers improved the slump of GC (from 110 to 210 mm) without significantly reducing the compressive strength (< 5 MPa). Water content of GC had a great

effect on the slump, but less so on the compressive strength. Increasing the GGBS content gradually decreased the slump but rapidly increased the strength, regardless of the curing temperatures of 20 or 75 °C. The GC with the minimum of 20% GGBS replacement achieved 33 MPa after 28 days curing at 10 °C. Air-dry curing provided a greater strength development of FA-based GC than water curing, though the opposite was observed for the 50/50 GGBS/FA GC. Consideration of these factors can significantly ease the manufacture of GC, enhancing its potential application in real structures, and consequently helping reduce global (CO₂) emissions.

Keywords

Geopolymer concrete; Alkali-activated materials; Fly ash; Ground granulated blastfurnace slag; Sodium hydroxide molarity; Mould release agent; Superplasticisers; Low temperature curing conditions; Slump; Compressive strength.

1. Introduction

Portland cement (PC), the primary binder to produce concrete, has become the most man-made material consumed worldwide with a total global production of 4.6 Gt/year [1]. In addition, cement production is estimated to be responsible for 5-8% of global total carbon dioxide (CO₂) emissions [2,3]. To try and address this issue, many approaches have been considered e.g. reducing CO₂ emissions from PC production by burning waste raw materials, increasing efficiency of the manufacturing process, and developing CO₂ capture and storage [4]; incorporating a higher percentage of mineral admixtures such as fly ash (FA) and ground granulated blastfurnace slag (GGBS) in blended PC [5,6]; and adopting alternative cement-like binders [7,8].

Alkali-activated materials (AAMs) are a promising alternative to PC. They are synthesised from the reaction product of an amorphous aluminosilicate-rich powder (binder) activated by an alkali metal source [9], which can create concrete with up to 45% less CO₂ than a typical PC-based counterpart [10,11]. The binder can come from natural sources such as metakaolin (MK) and pozzolan, or industrial by-products such as GGBS and FA, whilst the alkaline activators include alkali hydroxides, silicates and

carbonates [12]. It has been shown that alkali-activated concrete can have comparable mechanical properties to PC concrete, as well as improvements in resistance to fire and aggressive chemical attack [13,14].

Depending on the source of aluminosilicate, AAMs can be categorised into two key gel systems [9,15]. If geopolymers [16–18], the activation products of low-calcium binder sources including FA and/or MK are dominated by amorphous three-dimensional alkaline aluminosilicate hydrate (N-A-S-H) gels [19], the high-calcium sources such as alkali activated GGBS have the reaction products dominated by alkali charge-balanced aluminium-substituted calcium silicate hydrate (C-A-S-H) gels, which are similar to the C-S-H gels observed in PC [20,21]. In between these systems, the coexistence of N-A-S-(H) and C-A-S-H gels have been observed by alkaline activation of FA/GGBS [22,23] where the dominant product is strongly influenced by the FA/GGBS ratio. This study used the term geopolymer concrete (GC) because it focused on the slump and strength development of low calcium FA-based systems with up to 50% GGBS replacement, part of a larger research programme investigating the performance of the GC initially cured at a range of temperatures from 10 to 75 °C, to be published elsewhere [24,25].

In order to develop an optimal mix design for GC, the main factors which affect the mechanical strength development must be established. Many studies on GC, however, have concentrated on identifying optimal mix proportions based on the aggregate content, alkaline activator-to-binder ratio, molarity of sodium hydroxide, sodium silicate-to-sodium hydroxide ratio, superplasticizer-to-binder ratio, and water-to-solid ratio, and in particular the influence of curing temperature and duration [26–30]. Several guidelines have also been published [31–34]. However, other factors, regardless of the mix proportions, such as initial curing method by steam or oven [26], binder particle size [35–37], superplasticizer type [26,38–46], and in-air or -water curing [47] also have significant effects on the workability or strength development of GCs. These lack systematic investigation and were therefore investigated in this study.

More specifically, this study examined seven factors that influence the performance of GC (listed in the first columns of Table 2 in Section 2.2) including slump and strength development, as follows:

1. Initial curing conditions: Oven vs Steam;
2. Finest of FA: type N vs type S;
3. Types of superplasticiser: two naphthalenes (Conplast Sp430 and Daracem 215) vs two polycarboxylates (Viscocrete 10 and Glenium 51);
4. Water/solid ratios (with Alkaline/binder ratios of 0.3 ad 0.4)
5. FA replacement with up to 50% GGBS (with GGBS/binder ratios of 0.0; 0.1; 0.2; 0.3; 0.4; and 0.5), initially cured at 20 and 75 °C;
6. Initial curing at ambient temperatures of 10 (for at least 7 days) and 20 °C;
7. In-air or -water curing conditions until being tested after the initial curing at 20 or 75 °C.

Practical mould release agents were also trialled and a volume-to-mass conversion method was developed to identify the mass of solid sodium hydroxide dissolved into water at a specified molarity and ambient temperature to produce an 1-kg sodium hydroxide solution. The strength development of GGBS/FA GC cured at as low as 10 °C, has never been reported, but is important for the application of GC in temperate climates such as Europe.

2. Experimental details

2.1. Materials

The binders for the GCs were low-calcium FA and GGBS. The two FAs used had fineness categories S and N, which did not respectively exceed 12 and 40% by mass retained when sieved on a 0,045 mm mesh sieve. These, provided by CEMEX Ltd and conforming to BS EN 450-1 [48], differed only in fineness (Figure 1) as they were produced from the same located source and process. The GGBS was supplied by Hanson plc, to BS EN 15167-1 [49]. The chemical compositions by X-ray fluorescence (XRF) spectrometry are given in Table 1, and the particle size distributions by a Mastersizer 2000 are shown in Figure 1.

The alkaline activator was blends of sodium silicate and sodium hydroxide. The sodium silicate solution, commercially available as Crystal 100 with a specific gravity of 1.50 g/cm³, and containing 14

wt.% Na₂O, 28 wt.% SiO₂ and 58 wt.% water, was supplied by Tennants Distribution Ltd. The sodium hydroxide solution was prepared by stirring the Caustic soda pearls (solid NaOH), which was slowly added into the distilled water at a specific mass ratio calculated using the method recommended in Section 2.5. Four types of superplasticizer (SP) were investigated: two naphthalene types (Conplast Sp430 and Daracem 215) and two polycarboxylate types (Viscocrete 10 and Glenium 51). BASF Glenium 51 superplasticizer was selected to provide the necessary workability.

Table 1: Chemical composition and physical properties of FA type S and type N, and GGBS.

Oxide Composite	FA* (wt.%)	GGBS (wt.%)
SiO ₂	54.14	36.50
Al ₂ O ₃	26.65	11.60
CaO	3.36	39.46
Fe ₂ O ₃	6.51	0.43
Na ₂ O	1.07	0.39
K ₂ O	2.58	0.68
MgO	1.20	8.14
SO ₃	0.41	0.73
P ₂ O ₅	0.38	-
TiO ₂	0.60	0.84
L.o.I (wt.%)	3.10	0.03
Specific Gravity	2.265 (2.220)	2.880
Specific Surface Area (m ² /kg)	2770 (3320)	2390

Notes: * - FA type S, whereas the values of FA type N are in the parentheses.

Figure 2: Water absorption of fine and coarse aggregates from oven-dried to saturated surface-dry state.

2.2. Mix proportions

The mix design calculations for the GCs were based on Rangan [31], in which the density of the targeted GCs is assumed to be 2400 kg/m³ [26,27], and the mix proportions (ratios by mass) based on literature [26–29,53–56] were determined, including total aggregates/GC, fine aggregate/total aggregates, alkaline solution/(FA+GGBS), sodium silicate/sodium hydroxide, GGBS/(FA+GGBS), superplasticizer/(FA+GGBS). The actual amounts of raw materials for one cubic meter of GC were then calculated. Three curing temperatures of 10, 20 and 75 °C were selected. The mix proportion and curing conditions are presented in Table 2; the justification for the variables investigated are outlined at the end of section 1 (Introduction). According to this, each ID number from 1 to 7 listed in the rows in Table 2 represents a different factor investigated, such as the fineness of FA particles, inclusion of up to 50% GGBS, initial heat curing by oven or steam, long-term curing method by air-drying or in water, prolonged curing regime at a low temperature of 10 °C, and water-to-solid ratio (w/s).

FA type S was mainly designated for every mix except for those to investigate the effect of FA particle size, when FA type S was then replaced by type N. It is also worth noting that the w/s ratio in column (j) – Table 2 was calculated according to Eq. 1.

$$w/s = \frac{m_{water}}{m_{solid}} = \frac{m_{added_water} + m_{water_in_SS} + m_{water_in_SH}}{m_{FA+GGBS} + m_{solid_in_SS} + m_{solid_in_SH}} \quad Eq. 1$$

Where m_{water} is the total amount of water (kg); m_{added_water} is the extra water added during the casting phase of the GC excluding the amount of water absorption (Table 2) by the dried aggregates for 30 mins, (kg); $m_{water_in_SS}$ and $m_{water_in_SH}$ are the water contents within the sodium silicate and sodium hydroxide solutions respectively (kg). Meanwhile, m_{solid} is the total solid content (kg); $m_{FA+GGBS}$ is the mass of the binder (kg); and $m_{solid_in_SS}$ and $m_{solid_in_SH}$ are the masses of solid substance contained in the sodium silicate and sodium hydroxide solutions, respectively (kg).

From Table 2, Mix 1C could be considered as the standard/baseline/control mix design [26,27]; the others were determined by changing the mix design parameters of Mix 1C in columns (c), (e), (h) and (j), or the curing conditions in columns (t) and (u) to meet the requirements for each of seven factors set out in column (a). For example, compared to Mix 1C, Mixes 2A and 4B were identical Mix 1B had a reduction of the aggregates content (Agg/GC ratio – column (c)) from 0.77 to 0.70; Mixes 2B and 2C had 50% of GGBS replacement with FA (GGBS/binder ratio – column (h)) and were cured at 20 and 75 °C (columns (t) and (u)), respectively; and so on.

152 **Table 2: Mix design details.**

Factors	ID	Agg/ GC	Fine agg/ Agg	Alk/ Binder	SS/SH	NaOH solution (M)	GGBS/ Binder	SP/ Binder (%)	w/s	Quantity (kg)								Water for aggregates absorption (kg)	Added water (kg)	Initial Curing [27,29,56]		
		[26,28]	[27,28]	[26,27]	[26,28]	[53–55]	(h)	(i)	(j)	Coarse agg.	Fine agg.	Binder		NaOH solution	Sodium silicate	SP	(r)	(s)	(t)	Temp (°C)	Method	
										(k)	(l)	(m)	GGBS (n)									(o)
(a)	(b)	(c)	(d)	(e)	(f)	(g)	(h)	(i)	(j)	(k)	(l)	(m)	(n)	(o)	(p)	(q)	(r)	(s)	(t)	(u)	(v)	
Initial curing conditions: Oven vs Steam	1A	0.70	0.35	0.30	2.5	12	-	1.5	0.20	1092.0	588.0	553.8	-	47.5	118.7	8.3	13.6	24.5	24	75	Oven/Steam	
	1B	0.70	0.35	0.30	2.5	12	-	1.5	0.22	1092.0	588.0	553.8	-	47.5	118.7	8.3	13.6	36.0	24	75	Oven/Steam	
	1C	0.77	0.35	0.30	2.5	12	-	1.5	0.22	1201.2	646.8	424.6	-	36.4	91.0	6.4	15.0	27.3	24	75	Oven/Steam	
Finest of FA: type N vs type S	2A	0.77	0.35	0.30	2.5	12	-	1.5	0.22	1201.2	646.8	424.6	-	36.4	91.0	6.4	15.0	27.3	24	75	Steam	
	2B	0.77	0.35	0.30	2.5	12	0.5	1.5	0.24	1201.2	646.8	212.3	212.3	36.4	91.0	6.4	15.0	38.0	24	20	Amb.	
	2C	0.77	0.35	0.30	2.5	12	0.5	1.5	0.24	1201.2	646.8	212.3	212.3	36.4	91.0	6.4	15.0	38.0	24	75	Steam	
Two types of superplasticiser	3	0.77	0.35	0.30	2.5	12	-	1.5	0.24	1201.2	646.8	424.6	-	36.4	91.0	6.4	15.0	38.0	24	75	Steam	
W/s ratios: with alkaline/binder ratios of 0.3 and 0.4	4A	0.77	0.35	0.30	2.5	12	-	1.5	0.20	1201.2	646.8	424.6	-	36.4	91.0	6.4	15.0	19.0	24	75	Steam	
	4B	0.77	0.35	0.30	2.5	12	-	1.5	0.22	1201.2	646.8	424.6	-	36.4	91.0	6.4	15.0	27.3	24	75	Steam	
	4C	0.77	0.35	0.30	2.5	12	-	1.5	0.24	1201.2	646.8	424.6	-	36.4	91.0	6.4	15.0	38.0	24	75	Steam	
	4D	0.77	0.35	0.30	2.5	12	-	1.5	0.30	1201.2	646.8	424.6	-	36.4	91.0	6.4	15.0	66.5	24	75	Steam	
	4E	0.77	0.35	0.40	2.5	12	-	1.5	0.22	1201.2	646.8	394.3	-	45.1	112.7	5.9	15.0	5.4	24	75	Steam	
	4F	0.77	0.35	0.40	2.5	12	-	1.5	0.24	1201.2	646.8	394.3	-	45.1	112.7	5.9	15.0	15.4	24	75	Steam	
	4G	0.77	0.35	0.40	2.5	12	-	1.5	0.30	1201.2	646.8	394.3	-	45.1	112.7	5.9	15.0	42.8	24	75	Steam	
FA replacement with up to 50% GGBS, initially cured at 20 and 75 °C	5A	0.77	0.35	0.30	2.5	12	0.0	1.5	0.24	1201.2	646.8	424.6	-	36.4	91.0	6.4	15.0	38.0	24	20/75	Amb./Steam	
	5B	0.77	0.35	0.30	2.5	12	0.1	1.5	0.24	1201.2	646.8	382.2	42.5	36.4	91.0	6.4	15.0	38.0	24	20/75	Amb./Steam	
	5C	0.77	0.35	0.30	2.5	12	0.2	1.5	0.24	1201.2	646.8	339.7	84.9	36.4	91.0	6.4	15.0	38.0	24	20/75	Amb./Steam	
	5D	0.77	0.35	0.30	2.5	12	0.3	1.5	0.24	1201.2	646.8	297.2	127.4	36.4	91.0	6.4	15.0	38.0	24	20/75	Amb./Steam	
	5E	0.77	0.35	0.30	2.5	12	0.4	1.5	0.24	1201.2	646.8	254.8	169.8	36.4	91.0	6.4	15.0	38.0	24	20/75	Amb./Steam	
	5F	0.77	0.35	0.30	2.5	12	0.5	1.5	0.24	1201.2	646.8	212.3	212.3	36.4	91.0	6.4	15.0	38.0	24	20/75	Amb./Steam	
Initial curing at ambient temperatures of 10 (for at least 7 days) and 20 °C	6A	0.77	0.35	0.40	2.5	12	0.2	1.5	0.24	1201.2	646.8	315.4	78.9	45.1	112.7	5.9	15.0	15.4	168	10	Amb.	
	6B	0.77	0.35	0.40	2.5	12	0.2	1.5	0.24	1201.2	646.8	315.4	78.9	45.1	112.7	5.9	15.0	15.4	24	20	Amb.	
	6C	0.77	0.35	0.30	2.5	12	0.3	1.5	0.24	1201.2	646.8	297.2	127.4	36.4	91.0	6.4	15.0	38.0	24	20	Amb.	
In-air or -water curing conditions after the initial curing	7A	0.77	0.35	0.30	2.5	12	-	1.5	0.25	1201.2	646.8	424.6	-	36.4	91.0	6.4	15.0	42.5	24	75	Steam	
	7B	0.77	0.35	0.30	2.5	12	0.5	1.5	0.30	1201.2	646.8	212.3	212.3	36.4	91.0	6.4	15.0	66.5	24	20	Amb.	
	7C	0.77	0.35	0.30	2.5	12	0.5	1.5	0.30	1201.2	646.8	212.3	212.3	36.4	91.0	6.4	15.0	66.5	24	75	Steam	

Notes: Binder = (FA+GGBS); Agg/GC = total Aggregates/Geopolymer Concrete; Fine agg/Agg = Fine aggregate/total Aggregates; Alk/Binder = Alkaline solution/(FA+GGBS); SS/SH = Sodium Silicate/Sodium Hydroxide;

GGBS/Binder = GGBS/(FA+GGBS); SP/Binder = SuperPlasticizer/(FA+GGBS); w/s = water/solid; and Amb. = Ambient atmosphere.

2.3. Specimen preparation

2.3.1. Mould coating method

Mould release agents are normally applied to prevent concrete from adhering to the steel mould due to a thin layer of material with high lubrication properties (e.g. oil or wax) being applied before casting [57]. As a result, it makes demoulding easier and provides a smooth finished surface for the hardened concrete, as well as increasing the corrosion resistance of the steel mould. However, typical release agents commercially available for PC concretes are often inappropriate for GC. During some trials to produce the FA-based GC cubes, it was observed that despite commonly-used commercial release agents, for example Kingfisher and Sika release agents, being applied before the casting process, the GC adhered very strongly to the inner surfaces of the steel mould, so that the resultant struck cubes had either very rough finished surfaces (with aggregates often partially or completely exposed), or they were badly broken apart during demoulding. In addition, rust on the external surface of the steel mould was also developed due to steam-curing.

Unfortunately, no published studies have reported this issue in detail or with any substantial data, except for, for example, some general recommendations to apply “generally thick coatings of a viscous wax-based mould-release agent” [58] or a combination of various oils such as aliphatic compounds and waxes for the aluminium mould [59]. Therefore, an initial trial investigation was essential to develop an appropriate and effective release agent and coating process to produce samples efficiently as per the standardised requirements (BS EN 12390-1:2012 [60]) for mechanical and durability tests, together with a practicable demoulding process similar to that for PC concrete. Ten mould release agents were investigated (Table 3), including three designed for PC concrete with more specific properties closely related to the curing conditions of FA-based GC, as well as seven wax-based agents. Mix 1C was selected to investigate the effect of the release agents in Table 3 on 100 mm³ moulds and specimens steam-cured at temperatures from 40 to 100 °C and for 24 h.

179 **Table 3: Mould release agents**

Mould release agents from industry		Wax-based release agents	
Name	Properties	Name	Properties
Kingfisher mould release oil [61]	<ul style="list-style-type: none"> - A hydrocarbon and fatty acid blend - Used with metal or timber moulds - Resultant concrete has a smooth, hard, uniform finish with reduced incidence of blow holes 	NikWax Leather Cleaner; Cherry Blossom Wax & Shine; Neatsfoot Oil; Woly Shoe Wax; or Vaseline	<ul style="list-style-type: none"> - Blend of waxes and oils - Primarily used in the treatments of leather to replace the natural oil
Sika Release [62]	<ul style="list-style-type: none"> - A concrete release agent for precast or in-situ concrete - Used on steel, shuttering plywood, timber or plastic faced formwork - Improves surface finish and increases form work reuse 	Z30 Fluid [63]	<ul style="list-style-type: none"> - Good dewatering properties and penetration - Dry thin, flexible film doesn't attract dirt or dust - Not contain silicones or chlorinated solvents - Temperature: -20°C to +110°C
Sika Separol W [64]	<ul style="list-style-type: none"> - Smooth non-absorbent formwork - Absorbent formwork - Heated formwork (up to +70°C) - Textured formwork 	Waxoyl [65]	<ul style="list-style-type: none"> - Kills old rust and prevents new rust - Forms a flexible weatherproof skin - Apply to anywhere that water is likely to collect

180

181 As a result, the following coating method was developed based on the outcomes of the investigation
 182 in Section 3.1. Two layers, firstly Z30 Fluid and then Waxoyl, were applied to the moulds, with a
 183 minimum interval of 12 h for a natural drying of the first layer of Z30. Another minimum of 12 h was
 184 necessary for the second layer of Waxoyl to be naturally dried before casting the GC. The externally
 185 exposed part of the mould was then coated with Waxoyl before all steam curing at 30 °C or above.

186 2.3.2. Mixing procedures and curing conditions

187 The sodium hydroxide solution was produced in a fume cupboard as described in Section 2.1, which
 188 was then stored in an airtight container for at least 24 hours to allow it to cool down to room
 189 temperature (20 ± 2 °C) before slowly pouring into and mixing by hand with the sodium silicate to
 190 produce an activator solution. The alkaline activator was prepared at a designated mass ratio (SH/SS
 191 ratio in Table 2) at least 2 hours before mixing the concrete. Extra water and superplasticizer were
 192 then added to the blended activator solution. The extra water comprised the *added water* (column (s)
 193 in Table 2) calculated from the w/s ratio and the *water for aggregates absorption* (column (r))

determined from the 30-min water absorption of the aggregates to achieve the saturated surface-dry state (SSD).

All dry materials, including coarse and fine aggregates and binder (the FA, with or without GGBS), were thoroughly pre-mixed in a 35-litre laboratory pan rotary mixer for about three minutes until all the constituents were evenly distributed. The blended alkaline activator solution was then poured slowly into the running mixer for about a minute. The wet mixing was then continued for a further three more minutes. After conducting the slump test within the first 5 mins, the fresh GC was placed immediately into the prepared 100 mm³ moulds in at least three equal layers to reduce air voids entrapped within the GC more effectively. For each layer, the vibration table was run for a sufficient time to create a fully compacted concrete, typically between 15 seconds to several minutes at a frequency level of about 6/10. Care was taken to ensure that segregation and bleeding did not occur (i.e. coarse aggregate was still visible at the top surface). The moulds were then sealed with thin polythene sheets and immediately placed into an environmentally controlled room at 20 ± 2 °C and 65 ± 5 %RH for a maximum of one hour before initial curing. Depending on the designated initial curing regime, the specimens were heat-cured in an oven or a steam-curing tank for 24 h, or in an environmentally controlled chamber at 10 °C and 65 ± 5 %RH for a minimum of 7 days. After the initial curing period, the specimens were then stripped from the moulds, marked with ID numbers and placed in air in the environmentally controlled room at 20 ± 2 °C and 65 ± 5 %RH until being tested.

2.4. Fresh and hardened properties

A slump test complying with BS EN 12350-2 [66] was conducted once for every mix, immediately after mixing. Demoulded specimens were “as-received” samples [67] to record the masses and dimensions, which were used to calculate the volume and concrete hardened densities in accordance with BS EN 12390-7 [67]. All hardened properties were tested on 100 mm³ cubes. The compressive strength was measured conforming to BS EN 12390-3 [68], with a loading rate of 0.6 MPa/s. The water absorption was evaluated as a percentage of the difference in mass between oven dried and SSD cube specimens

after immersing in water to a constant mass, in accordance with ASTM C642 [69]. For each of these concrete hardened property tests, a mean of three measurements was reported.

2.5. Volume-to-mass conversion of sodium hydroxide solution at a specific molarity and temperature

In theory, to produce a litre of a sodium hydroxide solution having an N molarity, (M x N) grams of solid sodium hydroxide are dissolved into water (to generate exactly one litre of the solution), where M is the molar mass of solid sodium hydroxide. But it is very difficult to precisely measure the volume of a solution, in particular a large one. In practice, to avoid this issue, a percentage of Na₂O dosage was used instead to manufacture GC [70–73], leading to a complicated calculation of the alkaline modulus (Na₂O/SiO₂ ratio). A simpler method can be achieved by converting volume-to-mass of the sodium hydroxide solution as a mass measurement, which is always easier and highly accurate compared to that of a volume.

Many studies [29,54,55] have used sodium hydroxide molarities without any statement of how to precisely produce such solutions, whereas others [28,32,74] applied the converted values from volume-to-mass of sodium hydroxide as suggested by [26], but without verifying how they were calculated or measured.

In order to make the preparation process of sodium hydroxide solution more practical and accurate, a method was developed to describe the conversion calculation from volume-to-mass. To begin with, the concentration by mass of sodium hydroxide in a sodium hydroxide solution at T (°C) temperature can be defined and calculated according to Equation Eq. 2.

$$C_T(\%) = \frac{m_{NaOH_solid}}{m_{NaOH_solution}} \times 100 \quad Eq. 2$$

where, C_T is the concentration by mass of sodium hydroxide in the solution at T (°C) temperature (%mass/mass); m_{NaOH_solid} is the mass of the solid sodium hydroxide dissolved (g); and $m_{NaOH_solution}$ is the mass of the sodium hydroxide solution (g).

To produce V_0 (litres) of a sodium hydroxide solution with an N molarity, a mass of solid sodium hydroxide equal to $(M \times N \times V_0)$ grams is needed, where N is the molarity of the sodium hydroxide solution (mol/l); $M = 39.99711$ is the molar mass of solid sodium hydroxide (g/mol). Whereas the mass of this sodium hydroxide solution equal to $(D_T \times V_0)$ grams can be calculated using the solution density presented in Table 4, where D_T is the density of the sodium hydroxide solution (kg/l = 1000g/l) at T (°C) temperature. The Equation Eq. 3 presenting a series of hyperbola curves is then derived by substituting the two above parameters into Equation Eq. 2 with a specific molarity - N and at T (°C) temperature.

$$D_T = 3.99971 \times N \times C_T^{-1} \quad \text{Eq. 3}$$

Meanwhile, the relationship between density (D_T) and concentration (C_T) can be practically evaluated at a range of T from 0 to 100 °C from [75], which were selected and presented in Table 4 at more ambient temperatures of up to 40 °C, at which the manufacturing of GC is normally occurred. At each temperature, the density and the concentration by mass seem to have a linear relationship with a high coefficient of determination ($R^2 > 0.999$), which are represented in Eq. 4.

Table 4: Density of sodium hydroxide solution at 1 ATM [75].

Concentration-(C) (wt.%)	0 °C	10 °C	15 °C	18 °C	20 °C	30 °C	40 °C
	Density-(D) (kg/l)						
1	1.0124	1.0115	1.0107	1.0100	1.0095	1.0069	1.0033
2	1.0244	1.0230	1.0220	1.0213	1.0207	1.0177	1.0139
4	1.0482	1.0459	1.0444	1.0435	1.0428	1.0393	1.0352
6	1.0713	1.0683	1.0667	1.0656	1.0648	1.0609	1.0564
8	1.0943	1.0908	1.0889	1.0877	1.0869	1.0826	1.0780
10	1.1171	1.1132	1.1111	1.1098	1.1089	1.1043	1.0995
14	1.1624	1.1578	1.1555	1.1540	1.1530	1.1480	1.1428
18	1.2073	1.2023	1.1997	1.1982	1.1972	1.1918	1.1963
22	1.2519	1.2465	1.2439	1.2422	1.2411	1.2454	1.2296
26	1.2963	1.2906	1.2877	1.2860	1.2848	1.2789	1.2728
30	1.3400	1.3340	1.3309	1.3290	1.3279	1.3217	1.3154
34	1.3823	1.3760	1.3728	1.3708	1.3696	1.3632	1.3566
38	1.4234	1.4168	1.4135	1.4115	1.4101	1.4035	1.3967
42	1.4632	1.4561	1.4529	1.4508	1.4494	1.4425	1.4356
46	1.5018	1.4947	1.4911	1.4890	1.4873	1.4805	1.4734
50	1.5400	1.5326	1.5290	1.5268	1.5253	1.5181	1.5109

257

$$D_T = a_T \times C_T + b_T \quad \text{Eq. 4}$$

258

259 where, the slope-intercept, a_T , and the constant, b_T , are dependent on the ambient temperature as
 260 shown in Table 4. It is z when the concentration is zero, meaning that the solution is pure water: b_T is
 261 ideally equal to the water density. These parameters are determined at each temperature using linear
 262 regression (Table 5).

263 **Table 5: Parameters of Eq. 4 depending on the temperatures**

Temperature (°C)	0	10	15	18	20	30	40
a_T	0.0109	0.0107	0.0107	0.0107	0.0106	0.0106	0.0105
b_T	1.0083	1.0060	1.0045	1.0036	1.0030	0.9999	0.9960
CoD, R^2	0.9992	0.9993	0.9994	0.9994	0.9994	0.9993	0.9993

264

265 By respectively substituting values of the designed molarity and the environmentally controlled
 266 temperature into Equations *Eq. 3* and *Eq. 4*, the density and concentration would then be calculated.
 267 The results are shown in Table 6, based on which the converted mass of solid sodium hydroxide was
 268 determined for each unit mass of the solution and each unit mass of water at a specific temperature
 269 and designed molarity (Table 7). The range of sodium hydroxide molarity presented in this study (Table
 270 6) was collected from 4M to 18M because the majority of studies used sodium hydroxide solution with
 271 a molarity of 6 to 16 [26,28,32,54,55], whilst some tried 4M [29,76] or 18M [77]. Beyond this range,
 272 the impact of the sodium hydroxide solution would either be extremely low on the activation process
 273 (less than 4M) or become very corrosively aggressive for the users. However, the method proposed is
 274 straight forward and can be applied to identify the desired molarity with a concentration below 50%
 275 as shown in [75].

276

Table 6: Density and concentration of sodium hydroxide in the solution at a certain molarity and ambient temperature

Temperature (°C)	Parameter	Molarity							
		4M	6M	8M	10M	12M	14M	16M	18M
0	(1)	13.806	19.634	24.986	29.963	34.634	39.050	43.249	47.259
	(2)	1.159	1.222	1.281	1.335	1.386	1.434	1.480	1.523
10	(1)	13.860	19.719	25.104	30.113	34.817	39.264	43.493	47.534
	(2)	1.154	1.217	1.275	1.328	1.379	1.426	1.471	1.515
15	(1)	13.876	19.740	25.128	30.141	34.847	39.296	43.527	47.569
	(2)	1.153	1.216	1.273	1.327	1.377	1.425	1.470	1.513
18	(1)	13.886	19.752	25.143	30.157	34.865	39.315	43.547	47.590
	(2)	1.152	1.215	1.273	1.326	1.377	1.424	1.470	1.513
20	(1)	13.907	19.788	25.194	30.224	34.946	39.412	43.659	47.717
	(2)	1.150	1.213	1.270	1.323	1.373	1.421	1.466	1.509
30	(1)	13.940	19.831	25.245	30.281	35.009	39.479	43.730	47.790
	(2)	1.148	1.210	1.267	1.321	1.371	1.418	1.463	1.506
40	(1)	13.998	19.914	25.351	30.409	35.158	39.649	43.918	47.997
	(2)	1.143	1.205	1.262	1.315	1.365	1.412	1.457	1.500

Notes: (1): Concentration (%); (2): Density (kg/l)

Table 7: Converted mass of solid sodium hydroxide

Temperature (°C)	Type	Molarity							
		4M	6M	8M	10M	12M	14M	16M	18M
0	(A)	138	196	250	300	346	391	432	473
	(B)	160	244	333	428	530	641	762	896
10	(A)	139	197	251	301	348	393	435	475
	(B)	161	246	335	431	534	646	770	906
15	(A)	139	197	251	301	348	393	435	476
	(B)	161	246	336	431	535	647	771	907
18	(A)	139	198	251	302	349	393	435	476
	(B)	161	246	336	432	535	648	771	908
20	(A)	139	198	252	302	349	394	437	477
	(B)	162	247	337	433	537	651	775	913
30	(A)	139	198	252	303	350	395	437	478
	(B)	162	247	338	434	539	652	777	915
40	(A)	140	199	254	304	352	396	439	480
	(B)	163	249	340	437	542	657	783	923

Notes: Mass of solid sodium hydroxide in gram for (A) - 1 kg of sodium hydroxide solution; and (B) - 1 kg of water

Compared to the measurement from [26], there are about 10 grams or 4% difference, as shown in Table 8. In addition, this conversion will help specifiers avoid any complicated chemical calculations of sodium oxide mass ratio $M^+ = \frac{Na_2O}{binder}$ and alkali modulus $AM = \frac{Na_2O}{SiO_2}$ as suggested by [34,72,73,78,79].

Table 8: Comparison of the measurement of solid sodium hydroxide (in grams) for each kilogramme of solution between this study and [26]

Comparison	Temperature (°C)	Molarity				
		8M	10M	12M	14M	16M
Current study	20	251.9	302.2	349.5	394.1	436.6
	30	252.4	302.8	350.1	394.8	437.3
[26]	-	262.3	313.7	360.9	404.3	444.4

A 1-kg solution of sodium hydroxide for the conversion is recommended because there is a significant amount of heat emitted during the dissolution process leading to a large amount of water evaporated. The water can then be added up to produce a 1-kg solution when it has cooled down to the ambient temperature.

3. Results and Discussion

3.1. Efficacy of mould release agent

Three concrete industrial mould release agents and seven wax-based types were selected and tried using Mix 1C (see Table 3). The 100mm³ samples were steam-cured at 40 to 100 °C for 24 h before demoulding. The demoulding process and the surface state of the GC are compared with those of PC concrete and presented in Table 9.

299 **Table 9: Effect of release agent**

Name	Demoulding process	Surface state of GC specimen	Compared to PC concrete*
Kingfisher's Mould-Release Oil	- At 20 and 75 °C: very hard to clean with more than 30 minutes and much effort for each mould; GC adhered to the mould and broken apart at corners or edges.	- Broken concrete and rough finished surfaces with aggregates partially exposed.	Extremely difficult (impracticable)
Sika Release	- At 20°C: easier to clean compare with Kingfisher's, with about 15 minutes for each mould. - At 75 °C: more than 15 minutes for each mould and much effort was needed.	- Rough finished surfaces but no aggregates exposed.	Very difficult (extreme effort)
Sika Separol W	- At 75 °C: harder to clean than Sika Release with more than 15 minutes compared with.	- Rough finished surfaces but no aggregates exposed	Very difficult (extreme effort)
Cherry Blossom Wax & Shine	- At 75 °C: less effort to clean each mould compared with Sika Separol W with about 15 minutes.	- Rough finished surfaces but no aggregates exposed	Difficult
NikWax Leather Cleaner	- At 75 °C: easier to clean with much less effort than Sika Separol W with about 12 min for each mould.	- Normal finished surfaces	Not difficult
Neatsfoot Oil	- At 75 °C: a little bit easier than NikWax Leather Cleaner - At 20 °C: much harder to clean with 20 minutes for each mould because GC adhered to the mould and was broken at corners.	- Normal finished surfaces	Not difficult
Woly Shoe Wax	- At 20 and 75 °C: easier than NikWax Leather Cleaner and less effort clean with about 10 minutes for each mould.	- Normal finished surfaces	Quite easy
Vaseline	- At 75 °C: similar to NikWax Leather Cleaner - At 20 °C: in between Neatsfoot Oil and Woly Shoe Wax	- Normal finished surfaces	Not difficult
Z30 Fluid	- At below 50 °C: easy to clean with less than 10 minutes for each mould but still more effort than PC concrete. - From 50 to 75 °C: little paste adhered to the mould and needs more effort to clean with about 12 minutes for each mould. - From 75 up to 100 °C: GC adhered to the mould quite hard; needed a hammer and scraper to split the mould out of each other with hard strikes although 3 layers of Z30 has been coated before casting using heat gun to dry out.	- Normal finished surfaces at 75 °C and lower - Rough finished surfaces with aggregates partially exposed at above 75 °C	Quite easy
Waxoyl	- Similar to Z30 Fluid	- Similar to Z30 Fluid	Quite easy
A combination of Z30 Fluid and Waxoyl	- Much easier to clean the mould at all temperature with less than 10 minutes for each mould and a little bit more effort than PC concrete.	- Very good finished surface as PC concrete	Easy (Simple and appropriate for GC)

Notes: * - a reference for the demoulding process - as "easy" as for PC concrete when applying a conventional release agent

During the trial with the two commonly used mould release agents Kingfisher and Sika Release (Table 3), both of which provide an excellent finished surface for PC concretes, when applied to the GC, the

former caused the concrete to break and leave a rough finished surface with exposed aggregate. An extreme and impracticable effort using a hammer and chisel was also required to clean each steel mould due to the strongly adhered concrete (Table 9). The latter, Sika Release, was slightly better and did not result in broken concrete, but still left rough concrete surfaces with a very great effort required to clean the moulds. The trial also revealed that the elevated curing temperature at 75 °C caused the concrete to adhere more strongly to the steel mould compared with an ambient curing temperature of 20 °C. This indicates that the presence of alkali in the fresh concrete at high temperature might quickly damage the thin coating layer.

Other attempts were conducted, taking into account the effect of the elevated curing temperature and recommendations of using wax-based agents (Table 3), including Sika Separol W - a mould release agent suitable for concrete with heated formwork up to +70 °C; leather treatment waxes, spray-applied in layers (NikWax Leather, Cherry blossom Wax & Shine, Neatsfoot Oil, Woly Shoe Wax, and Vaseline); and corrosion protection waxes (Z30 Fluid, a heavy duty waxy film which manufacturer claims works across a temperature range from -20 to 100 °C, and Waxoyl, which creates a waxy waterproof film). Sika Separol was no better than Kingfisher or Sika Release. The leather treatment waxes exhibited a significant improvement in providing a smooth finished surface without exposing the aggregate. However, a significant effort was still required to clean the mould (more than 10 minutes per mould). The most effective leather treatment wax was the Woly Shoe Wax, although a minimum of 3 layers is recommended, with which the effects of both Z30 Fluid and Waxoyl were comparable.

Finally, instead of applying two or three layers of each agent, a combination of one layer of Z30 Fluid and then another layer of Waxoyl was investigated, with a 12 h gap for each layer to dry naturally. An excellent smooth finished surface was observed on demoulding, and a minimal effort to clean the mould was required, similar as for typical PC concretes. Although the underling chemical or physical reasoning for this enhanced performance is beyond the scope of this study, the authors highly

recommend these materials and this process for use with casting GCs. The 12 h intervals for a natural drying can be reduced by an accelerated drying process.

3.2. Ease of placement and compaction of fresh GC

Placement (workability) of fresh GC is more difficult than PC concrete of equivalent slump [27,80] due to the high viscosity of the alkaline activators, owing mainly to the sodium silicate solution. The same conclusion was illustrated in this work as the fresh GC was highly cohesive, and became relatively stiff, especially when the w/s ratio was lower than 0.24. Mix 1C (Table 2) was compared with a PC concrete mix with the same aggregate content and a water-to-cement ratio of 0.6. They both resulted in the same slump of 30 mm, but the PC concrete was much easier to place than the stiffer and more cohesive GC. This was also observed during the compaction process. If the PC concrete needed about 30 seconds on the vibration table at an amplitude of 4/10 for full compaction without segregation, the equivalent GC Mix 1C required more than 30 *minutes* vibration at an increased amplitude of 6/10. Amplitude here is a relative value, and relates to the vibrations/movements of the vibrating table over time (e.g. per second), rather than actual distance moved.

In addition, entrapped air in PC concretes, which contributes to the total voids that negatively affect the compressive strength, can be minimized with an optimum water content for a given compaction method [81]. However, due to high viscosity of activators containing a highly soluble silicate [27], the removal of air voids entrapped within the GC during the casting process requires greater energy inputted into the compaction process. i.e. a greater amplitude of vibration for a longer duration, in particular for low slump mixes [58]. For example, Mix 2A in Table 2 with the w/s ratio of 0.20, bubbles kept coming up to the cast surface after vibrating for more than 20 minutes at an amplitude of 6/10 (although the number of bubbles reduced with time). This mix did not experience any sign of segregation after more than 30 minutes of compaction. When the w/s ratio increased to 0.3 (Mix 2D) and the amplitude reduced to 4/10, bubbles still appeared after segregation had occurred. This indicates that, unlike PC concrete, compaction by means of a vibration table might not effectively remove all the air-entrapped voids before segregation begins, meaning that compaction is

inconsistent even for the same mix design, which could encourage misjudgements in the comparisons of the performance of hardened GCs, e.g. the compressive strength and/or porosity.

3.3. Influence of investigated factors on slump and compressive strength development of GC

When the GC was cured at 75 °C and above for at least 24 hours, the 7-day compressive strength was used for analysis instead of 28 days. This is based on the comparisons presented in Table 10, in which more than 93% of the 28-day compressive strengths were achieved at 7 days, regardless of FA type, heat curing method, or GGBS inclusion (or not). A similar observation of FA-based GC was also reported [28]. The 28-day strengths were reported only when the water absorption values were measured, or when the strength development over time from 7 to 28 days was discussed, or when the GCs were initially cured at ambient temperatures (10 or 20 °C). It is worth noting that although the designed density of the GC was 2400 kg/m³, the actual measured densities were in the lower range of 2250 to 2410 kg/m³. These lower values might be due to the air-dry curing period right after demoulding.

Table 10: Ratios of 7- and 28-day compressive strength

Mix ID	Slump (mm)	Curing			Age (days)	Density (kg/m ³)	Compressive strength (MPa)				7/28-day C.S. ratio (%)
		Time (h)	Temp (°C)	Method			S1	S2	S3	Mean	
1A	200	24	75	Oven	7	2270	47.8	45.7	46.1	46.5	95.8
					28	2250	47.7	49.9	48.1	48.6	
1B	220	24	75	Steam	7	2260	44.7	46.0	45.3	45.3	93.2
					28	2230	49.0	48.4	48.5	48.6	
1C	30	24	75	Steam	7	2330	49.1	49.5	47.4	48.7	99.2
					28	2310	50.9	49.1	47.2	49.1	
2A*	50	24	75	Steam	7	2310	37.5	38.7	38.2	38.2	96.4
					28	2300	38.8	40.2	39.7	39.6	
2C*	10	24	75	Steam	7	2410	104.5	104.5	105.0	104.6	96.9
					28	2390	108.3	107.6	108.2	108.0	
4E	30	24	75	Steam	7	2350	69.5	68.0	69.2	68.9	97.2
					28	2330	69.9	71.7	71.1	70.9	

Notes: C.S - Compressive Strength;

* - type N of FA;

S1 – S3 - tested sample 1 to 3.

3.3.1. Oven- and steam-cured methods

Mixes 1A, 1B and 1C were initially cured by both oven and steam methods at 75 °C for 24 h. The slump, density and compressive strength are shown in Table 11. Mix 1C was selected as the standard/control

mix design based on the recommendations from [26,27], and Mix 1B was designed to have a reduction of Agg/GC ratio from 0.77 (for Mix 1C) to 0.70 following suggestion by [28]. Mix 1A then had a reduction in w/s ratio from 0.22 to 0.20 compared to Mix 1B, the w/s ratio thus being similar to the mixes investigated by [26].

Compared with the oven, the steam curing resulted in equivalent or higher compressive strengths of up to 15% at 7 days, and 20% at 28 days for Mix 1A, as discussed below. This large difference in compressive strengths between the two curing methods compared to those of Mixes 1B and 1C was the reason for only measuring the water absorption at 28 days for Mix 1A. The results however, showed similar values: 4.6% by oven and 4.5% by steam.

Despite having the same w/s of 0.22, the significant increase in slump from 30 mm of Mix 1C to 220 mm of Mix 1B can be explained by the increase of the paste content by 7%, which not only directly reduced the overall viscosity of the fresh GC but also increased the absolute water content by 32%. In addition, the difference in compressive strength of the steam and oven methods increased from approximately 0 to 12%.

Table 11: Oven- and steam-cured methods of the FA-based GCs

Mix ID	Slump (mm)	Curing method	Age (days)	Density (kg/m ³)	Compressive strength (MPa)				Water absorption (%)
					S1	S2	S3	Mean	
1A	200	Oven	7	2270	47.8	45.7	46.1	46.5	-
			28	2250	47.7	49.9	48.1	48.6	4.6
		Steam	7	2310	55.6	52.3	52.2	53.4	-
			28	2310	59.2	57.4	58.6	58.4	4.4
1B	220	Oven	7	2260	39.6	41.6	40.7	40.6	-
		Steam	7	2260	44.7	46.0	45.3	45.3	-
1C	30	Oven	7	2330	48.7	49.3	48.5	48.8	-
		Steam	7	2320	49.1	49.5	47.4	48.7	-

Notes: S1 – S3 - tested sample 1 to 3.

However, the advantage of the steam over the oven curing on the compressive strengths (as shown in Table 11) contrasts with the observations of Hardjito and Rangan [26], in which the compressive strength of samples with a similar mix proportion cured in an oven were approximately 15% higher than that cured by steam. This discrepancy was possibly because at a very low water content of 20 to 22% (of the total solids) with less than 5% chemically bound in the gel structure of an N-A-S-H system

[82], it is more crucial to maintain a sufficient amount of water available within the concrete as a medium for the dissolution stage of aluminosilicate particles and transferring ions, and oligomers synthesised, in which the steam curing provided some advantage. It is therefore unlikely to hinder the polycondensation stage occurring at higher w/s ratios [83]. Another factor might also be due to the difference between the initial curing temperatures of 60 °C by [26] and 75 °C in this research, according to which the rate of strength development could increase by up to 10 times [84].

Fineness of FA

Figure 3 illustrates the influence of the FA particle size (represented as type N (coarser) and type S (finer) and conforming to BS EN 450-1 [48]), on the 7- and 28-day compressive strengths of Mixes 2A, 2B and 2C. It is worth noting that Mixes 2B and 2C were identical, except for being initially cured at 20 and 75 °C, which were designed to have 50% replacement of GGBS with FA compared to Mix 2A. In addition, they have a higher w/s ratio of 0.24 to achieve a similar workability. These mixes were therefore selected because they were at the boundary of the mix range from FA-based GC to 50/50 GGBS/FA GCs.

The compressive strength of both FA types increased with age up to 28 days depending significantly on the curing temperature and GGBS content. The compressive strengths of the FA-based GC using type S were 20% higher than those using type N at the same age (Figure 3a), consistent with the findings of others [35–37], which showed that finer particles of FA improved the compressive strength. This fineness effect however, was negligible, regardless of the curing temperature of 20 or 75 °C when 50% of FA was replaced by GGBS (Figure 3b and c); the heat curing at 75 °C provided a much higher early age strength at 7 days than those cured at an ambient temperature of 20 °C, similar to the observations in Table 10. An inverse trend was observed for the slump, increasing from 30 to 50 mm for FA-based GC when the fineness decreased, i.e. from type S to type N; whereas the slump stayed at 15 to 20 mm with 50% GGBS, regardless of the FA particle size.

Figure 3: Effect of FA type N and S on the compressive strength of GCs containing a) 100% FA cured at 75 °C for 24 h; b) 50/50 GGBS/FA cured at 20 °C; and c) 50/50 GGBS/FA cured at 75 °C for 24 h.

Superplasticizer

As observed and discussed in Section 3.2, the workability of GC is considerably lower than PC concrete with the same w/s ratio due to the high viscosity of soluble silicate in the activator compared with water. In addition, to achieve a compressive strength of 35 MPa and above, the w/s ratio of GC might need to be kept as low as 0.22 [26], leading to a significant decrease in the workability. An appropriate high-range water reducing admixture – superplasticizer (SP) – is therefore essential to overcome this issue without increasing the amount of water in the mix design, which would increase the porosity of the binder and hence reduces the mechanical strength. However, because commercially available SPs are currently designed for PC and their variants with additions of mineral admixtures such as FA, GGBS and silica fume, they can have unexpected effects on GC [85]. Apart from the physical properties and chemical composition of FA and GGBS, in which particles' surface-absorbent capability is different from that of PC, the high alkaline environment (i.e. pH > 13) induced by the activators prompts a chemically unstable state for most types of SP, except for the naphthalene-based group [38]. Comprehensive reviews of additives for alkali-activated GGBS [86] and geopolymer [87] was reported by Rashad; he concluded that naphthalene-based SPs improved both workability and compressive

strength of the alkali-activated GGBS depending on activator type and concentration, whereas Sika Viscocrete-3430, a modified polycarboxylate, was recommended for FA-based geopolymer.

Table 12 summarises from literature the effects of five typical commercial SPs, including naphthalene-based, modified polycarboxylate, liginosulfonate, vinyl copolymer, and melamine, on the workability and compressive strength of both GGBS- and FA-activated GCs. The ‘positive’ effect of the SP on the workability or compressive strength was considered when the former increased effectively, or the latter showed no significant degradation. ‘Positive’ marks (‘+’) for both properties indicate an appropriate SP. In other words, the recommended SP has to improve the workability without any negative impact on the compressive strength. According to Table 12, it seems that naphthalene-based SPs are better suited for both alkali-activated systems, whilst the latest generation of modified polycarboxylate SPs exhibited a ‘positive’ effect for only FA-based geopolymer.

Table 12: Effects of typical superplasticizers on alkali-activated GGBS and geopolymer

Concrete type	Superplasticizer type	Workability		Compressive strength	
		“+”	“-”	“+”	“-”
Alkali-activated	Lignosulfonate	[39]	[40]; [41]		[40]; [41]; [39]
	Naphthalene	[39]; [38]; [42]	[40];	[38]	[40]; [39]
GGBS	Vinyl copolymer		[43]; [38]; [42]	[38]	[43]
	Polycarboxylate	[44]	[43]; [38]; [42]	[43]; [38]	
FA-based	Melamine		[38]; [42]	[38]	
	Lignosulfonate		[45]		
	Naphthalene	[26]; [46]	[88]	[26]; [46]	[88]
	Vinyl copolymer		[43]	[43]	
	Polycarboxylate	[45]; [46]	[43]; [88]	[43]	[88]; [46]
	Melamine		[45]		

Notes: Workability “+”: increase Compressive strength “+”: no decrease or increase

"-": no change or decrease "-": substantial degradation

In addition, Jang *et al.* [76] recently studied the fresh and hardened properties of blended GGBS/FA geopolymer pastes incorporating naphthalene-based and polycarboxylate-based SPs. They investigated GGBS/(FA+GGBS) ratios of 0, 0.3, 0.5, 0.7 and 1, with SP dosages of 0, 1, 2, 3, 4%, cured at room temperature. They found that an increase in GGBS content led to a decrease in the workability for both SP types, when 4% was added. It was also observed that at the GGBS/(FA+GGBS) of 0.5, and by increasing the dosage of SP, the workability increased, but more effectively using polycarboxylate-

based SPs. However, a degradation in compressive strength was also observed when more than 1% polycarboxylate-based was added.

Based on the above reviews, four SPs from a variety of manufacturers were selected for investigation in Mix 3: two naphthalenes (Conplast Sp430 and Daracem 215) and two polycarboxylates (Viscocrete 10 and Glenium 51). The mix proportions are shown in Table 2 and the measured compressive strengths, slumps and water absorption are presented in Table 13. Compared to the control mix without SP, the slumps of the mixes increased with all SPs by 60 to 100 mm from 110 mm, whilst their compressive strengths provided a slight variation within 5MPa. The polycarboxylate group showed some increases in both workability and early compressive strength (compared to the naphthalene), which confirmed the recommendations by Rashad [87] discussed in section 3.3.3. However, regards the GC using Viscoscrete 10, although it had the highest slump, the 28-day compressive strength was about 10% less than that of the same polycarboxylate type – Glenium 51. The Glenium 51 therefore, with the highest compressive strength (with a small variance), an average slump and the lowest porosity (Table 13), is recommended.

Table 13: Effect of naphthalene and polycarboxylate SPs on GC.

Mix ID	SP type	Slump (mm)	Age (days)	Hardened Density (kg/m ³)	Compressive strength (MPa)				Water absorption (%)
					S1	S2	S3	Mean	
3	No SP	110	7	2330	34.6	33.9	34.3	34.2	-
			28	2300	35.2	34.5	35.2	35.0	4.5
	Conplast Sp430 (Fosroc)	170	7	2310	32.5	33.7	32.6	32.9	-
			28	2290	34.0	33.2	33.7	33.7	4.4
	Daracem 215 (Grace)	170	7	2300	30.8	29.1	30.2	30.0	-
			28	2300	31.2	31.2	31.2	31.2	5.5
	Viscocrete 10 (Sika)	210	7	2340	33.2	35.0	34.5	34.2	-
			28	2270	36.0	29.7	33.1	32.9	4.7
	Glenium 51 (BASF)	180	7	2330	36.4	36.1	36.3	36.3	-
			28	2290	36.2	36.5	36.4	36.4	4.3

Notes: S1 – S3 - tested 1 to 3.

3.3.4. Water-to-solid (w/s) ratio

Figure 4 illustrates the relationship between the w/s ratio and the compressive strength of the FA-based GCs with Alk/FA ratios of 0.3 and 0.4. Increasing the w/s ratio resulted in a decrease in the

compressive strength and an increase in slump, similar to the PC concrete, and consistent with others [26,28,74]. It is also clear that increasing the Alk/FA ratio from 0.3 to 0.4 improved the compressive strength of the FA-based GCs by 1.5 to 2 times and became more linear with the w/s ratio from 0.20 to 0.30, which is in good agreement with [28]. It is worth noting that within such a small range of w/s ratio (from 0.20 to 0.30), the difference in slump was significant, from 10 mm (S1) to 270 mm (S5a) [81,89]. A virtually identical trend of non-linear relationship (i.e. an S curve) between the slump and the w/s ratio was recorded (Figure 4). The slump showed a slight reduction when the Alk/FA ratio increased with a w/s of 0.24 and above. This might be due to the increase of the highly viscous activator content, which consequently reduces the amount of added water (Table 2) for the same w/s ratio. Both of the Alk/FA ratios exhibited a sharp increase in slump when the w/s ratios raised narrowly from 0.22 to 0.24. Outside this range however, the slump gradient reduced. This indicates that the slump of FA-based GC is sensitive to a small change of w/s ratio, within certain ranges. In addition, the water absorption of the mix designs containing a lower Alk/FA ratio of 0.3 rose linearly, as expected, with an increase of the water content, i.e. the porosity, similar to PC concrete.

Figure 4: 7-day compressive strength and slump versus w/s ratio of mixes 4A to 4D for Alk/FA=0.3; and 4E to 4G for Alk/FA=0.4, plus water absorption versus w/s ratio for the mixes with Alk/FA ratio=0.3.

3.3.5. FA replacement with GGBS

The effect of up to 50% FA replacement with GGBS on compressive strength and slump of GCs cured at ambient (20 °C) and elevated temperature (75 °C) are shown in Figure 5. It can be observed that a higher content of GGBS leads to 1) an increase in compressive strength regardless of the curing temperature, consistent with [23,73,80,90,91], and 2) a gradual decrease in the workability, similar to observations from [80,90,92]. When the blended GCs were cured at 20 °C, the GGBS/binder ratio in a range from 0 to 0.4 demonstrated a linear relationship with the 28-day compressive strength, corresponding well with Nath and others [80,90] using up to 30% GGBS replacement. With 40 to 50% GGBS in the binder, the increase in the compressive strength became minimal (Figure 5). This suggests that at ambient temperatures, the GGBS rather than the FA contributed significantly to the strength development of the GC. When the GGBS content increased from 0 to 40% or higher, the resulting compressive strength increased from 20 to 80 MPa. Of the blended GCs cured at 75 °C for 24 h, the 7-day compressive strength rose rapidly from 35 MPa to more than 85 MPa when the GGBS/binder ratio increased from 0 to 20%, beyond which a slower increase was recorded. A similar trend has also been reported for the binder [93] or mortar [73]. In addition, when more than 20% GGBS was added (mixes 5D to 5F, Table 2), the freshly blended GCs set quickly, which consequently might affect the slump measurement of the FA/GGBS blended GC if the testing process was delayed. These observations are in agreement with generally accepted findings that the setting time of GC reduces rapidly with an increase in GGBS content [76,80,92].

Figure 5: 7- and 28-day compressive strengths and slump versus GGBS/binder ratio of blended GCs cured at 20 and 75 °C, respectively.

3.3.6. Curing at ambient and low temperatures

Figure 6 presents the compressive strength development of two blended GC mixes with minimal GGBS content to binder of 20 and 30% and initially cured at ambient (20 °C) or low (10 °C) temperature for up to 28 days. They were selected because the inclusion of GGBS is a possible solution to accelerate (without using heat curing) the hardening process of FA-based geopolymer, which is very slow (in weeks rather than days) at ambient temperature [26,94,95]. Three sealed curing methods were used (see section 2.3.2), including:

- Method 1 – samples sealed and cured at 10 or 20 °C all the time;
- Method 2 – samples sealed and cured at 10 °C for 7 days, before air-dried at 20 °C; and
- Method 3 – samples sealed and cured at 20 °C for 24 h, before air-dried at 20 °C.

Figure 6: Compressive strength of: a) Mix 6A (curing method 1 and 2), 6B (curing method 3); and b) 6C (curing method 1 and 3).

Figure 6a shows that after being sealed and cured at 10 °C for 7 days using method 1 or 2, the compressive strength of the 20% GGBS mix (Mix 6A or 6B) was very low and achieved only about a third of that cured at 20 °C by method 3 (Mix 6B). However, by continuing with the air-cured condition at 20 °C for a further 21 days with method 2, resulted in a comparable 28-day compressive strength to method 3, which was nearly twice as much as with method 1. It is clear that the prolonged curing at a low temperature of 10 °C has considerably retarded the polycondensation process of the blended GC, whereas the air-dried period at 20 °C is more appropriate for accelerating the reaction kinetic that contributes primarily to the strength development. When curing took place at 20 °C with methods 1 and 3 (Figure 6b), the 7-day compressive strengths were comparable, whilst at 28 days, the strength gained by method 1 was slightly higher. Method 3 however, is more applicable for placing insitu concrete in temperate climates.

3.3.7. Air dry curing vs water curing of GCs initially cured at 20 and 75 °C

The effect of curing in air or water after the initial 24 h curing at either 20 or 75 °C was different for the FA-based (Mix 7A) and FA/GGBS blended (Mixes 7B and 7C) GCs, as shown in Figure 7. It is worth noting that Mixes 7A, 7B and 7C were identical to Mixes 2A, 2B and 2C respectively but at higher water

contents in order to increase the workability. For the FA-based GC initially cured at 75 °C for 24 h and then maintained in water at 20 °C until tested, the results illustrated a marginal increase in the compressive strength between 28 and 91 days, which was 15% less than the 28-day compressive strength of the same mix design maintained in air at 20 °C and 65 %RH (Figure 7a). These observations are in agreement with findings based on mortars [47], but the strength development rate of the GC in water in this research was much lower. The presence of water in the pore structure of the N-A-S-(H) gel could explain the differences between the air-cured and water-cured conditions presented here. As described by Duxson *et al* [96], the polycondensation process releases water, which therefore, as a catalyst or retarder, would be hindered if the pore solution is saturated, or facilitated if it is dried. The 50/50 GGBS/FA GC showed the opposite trend however, in which prolonged curing in water improved the 28-day compressive strength by more than 20% compared with the air-dried curing condition, although the mix contained a high w/s ratio, which was equivalent to the strength of Mix 7C cured in air after initial curing at 75 °C for 24 h (Figure 7b). It seems that with a 50% GGBS substitution, the C-N-A-S-H gel containing a high bound water content becomes predominant [22,97], which consumes more water for the ongoing gel evolution (and thus strength development) of such a high calcium system. In addition, there were differences between the densities of the GC cured in water and in air, greater for FA-based GC (Mix 7A) of 2360 and 2250 kg/m³ and less for 50/50 GGBS/FA GC of 2390 and 2350 kg/m³, respectively. However, the water-saturated densities of GCs are comparable to that of OPC concrete. This again confirmed that the assumption in the mix design calculations was appropriate.

Figure 7: Effect of curing conditions on the compressive strength of GCs with a) 100% FA and b) 50% GGBS.

4. Conclusions

Experiments were conducted to systematically investigate the following factors influencing the slump and strength development of FA-based GC with a substitution of up to 50% GGBS: FA particle fineness, FA replacement with GGBS, superplasticiser type, initial curing condition (steam or oven), curing condition (air or water), initial curing temperature (10, 20 and 75 °C), and the water-to-solid ratio. Suitable mould release agents were trialled and identified, and a volume-to-mass conversion method of sodium hydroxide solution at a specific molarity and temperature was developed. The following conclusions can be drawn:

- i. A volume-to-mass conversion of sodium hydroxide solution was developed based on the designed molarity and the ambient temperature at which the GC is cast, where the results are within 4% compared to literature [26]. The converted masses of solid sodium hydroxide for 1 kg solution or water are presented in Table 6, in which the use of 1 kg solution is recommended because the evaporated water by heat during the dissolving process can be balanced out when the solution cools down to ambient temperature.

- ii. The mould release agents available for PC concrete are mostly inappropriate for GCs because they caused rough finished surfaces, sometimes with exposed aggregate, as well as requiring extreme effort to clean the steel moulds when de-moulding. A combination of Z30 Fluid and Waxoyl (wax-based agents) is recommended for GC, providing an excellent finished surface with minimal cleaning effort, similar to PC concrete.
- iii. The ease of placement of GC is considerably lower than comparable PC of a similar slump. Mechanical vibration removes a significant amount of the air entrapped within the GC due to its high viscosity. However, the effectiveness of this vibration decreases as the w/s ratio increases because the entrapped air bubbles are released continuously even after segregation has occurred. This might cause inconsistencies in compaction of GC leading to an adverse influence on consequent mechanical and durability properties.
- iv. FA-based GC initially steam cured at 75 °C for 24 h enhanced the compressive strength by up to 20% compared to that cured in an oven at the same temperature, especially at a w/s ratio as low as 0.22.
- v. When changing the FA from type N to S, the benefit of a finer binder on the compressive strength of GCs decreased as the GGBS content increased (a 20% difference for 100% FA to almost zero with 50% GGBS replacement).
- vi. Both naphthalene and polycarboxylate types of superplasticizer improved the slump of the GC. However, a superplasticizer such as Glenium 51, might be beneficial for both compressive strength and water absorption.
- vii. An increase of w/s ratio, i.e. water content, from 0.2 to 0.3 decreased the compressive strength of the FA-based GC approximately linearly with alkaline-to-binder ratios of 0.3 and 0.4. It increased the water absorption however, of the FA-based GC with an alkaline-to-binder ratio of 0.3. The slump increased with the increase in the w/s ratio regardless of the alkaline to binder ratio; however, a sharp rise in slump occurred in a small range of w/s ratios from 0.22 to 0.24. Outside this range, the effect of additional water quickly becomes marginal.

- viii. Increasing the GGBS content in the GCs significantly increased the compressive strength regardless of curing temperature of 20 or 75 °C, but it slightly decreased the slump of the same w/s ratio mixes. At 20 °C, the increase in compressive strength became negligible when the GGBS content was beyond 40%, reaching 80 MPa after 28 days, whereas a similar change occurred when the GC was substituted by 20% GGBS and cured at 75 °C.
- ix. Having retarded considerably the polycondensation process of the 20% GGBS GC in a prolonged curing process at 10 °C, a moderate 28-day compressive strength of 33 MPa was achieved.
- x. Between the air-dry and water-cured methods, the former improved the compressive strength development of the FA-based GCs whereas the latter enhanced the blended GC with up to 50% GGBS.

These findings contribute to the understanding of GC systems including the practical knowledge required for the increased industrial application of GCs, enhancing the potential for the increased application of GC in real structures, and consequently helping reduce global (CO₂) emissions. They also contribute into the later phases of our research programme, reported elsewhere [24,25].

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