Effect of discontinuous curing and ambient temperature on the compressive strength development of fly ash based Geopolymer concrete

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Abstract. In the current study, 6 mixtures of Geopolymer concrete have been studied. The effect of discontinuous curing in oven and in atmosphere ambient temperature has been inspected by exposing the Geopolymer concrete specimens to temperature in the oven for few hours then stopping the oven to let it cool down to the ambient temperature. The compressive strength test of 100x200 mm cylindrical specimens for each mixture has been performed at different ages. It was found that the ambient temperature has vast effect on the compressive strength of the Geopolymer concrete in discontinuous curing. Discontinuous curing saves energy and can be a good replacement to the continuous curing when the ambient temperature is 40° C or more. Specimens that were cured continuously in ambient temperature of 43° C has resulted in compressive strength of 23 MPa at age of 40 days; to enhance the compressive strength, it is advised to impose few hours of discontinuous oven curing. It was also found that the rest period (starts when pouring concrete in forms and ends when imposing oven curing to the Geopolymer) has a positive influence on the compressive strength of Geopolymer concrete, but when no rest period is allowed, the later ages compressive strength is remarkably higher.

1 Introduction

Geopolymer concrete or so-called mineral-polymer concrete is one of the promising innovations in the concrete industry [1]. It has mainly attracted the attention of the scientists and the concrete industry entities as an alternative to conventional concrete because of being much more environmental friendly than the latter. Large amount of carbon dioxide is being released into the air during the manufacturing of Portland cement [2, 3]. For producing one ton of Portland cement, one ton of Carbon dioxide is emitted to the atmosphere [4]. Knowing that concrete is the second most consumed material, just below water, with an annual consumption of (4180) metric tons/ year (as for 2014) [5]. The result is (4180) metric tons/ year of Carbon dioxide is being added to the atmosphere every year from the production of Portland cement that will be used for the manufacturing of the conventional concrete.

Portland cements paste gains strength from the process of hydration that results in C-S-H gel. The mechanism of developing strength in Geopolymer is explained by the poly-condensation of the precursors of silica and alumina.

Mineral-polymer results from the polymerization of materials that alumina and silica constitutes most of its contents. To enforce the polymerization of the alumina-silica enriched materials, an alkaline activator such as sodium hydroxide and sodium silicate solution is to be added. The addition of the alkaline solution results in the dissolving of the oxide minerals from the silica-alumina

oxides and finally poly-condensation to form the final stable silico-aluminate product [6]. The production of the Geopolymer can be as described below:

In spite of the fact that since Davidovits has published his innovations in Geopolymer binders and cements and many researchers have studied the physical and chemical properties of the Geopolymer and the production of it for various uses, none of the concrete standardization entities have adopted standard procedures for producing Geopolymer concrete. Thus, more information are required to provide more knowledge for the database in the field to be enriched with more aspects resulting in better understanding of the physical and chemical properties that contribute to the Geopolymer concrete properties.

The advantages of Geopolymer concrete to the environment and the durability of it are considered much higher than those of the Portland cement concrete and make it a good replacement to the conventional Portland cement.

One of the innovative developments in concrete industry is the high range water reducing agents (HRWRA) which is necessary for workable and durable concrete. HRWRA enhance the fresh properties of the concrete, and improve hardened concrete properties,

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particularly, the durability of the hardened concrete [7-11].

Due to the advantages of the HRWRA to the industry of concrete, the Geopolymer concrete, as a potential alternative to Portland cement concrete, needs to be investigated for the appropriate type of HRWRA for Geopolymer concrete. The current study used naphthalene-based superplasticizer to get the required workability and to enhance the compressive strength of the Geopolymer concrete.

2 Experimental Work

For the production of the Geopolymer for the research program, the following materials were used: fly ash class F, sodium hydroxide flakes, sodium silicate liquid, crushed gravel, sand, superplasticizer, and tap water.

2.1 Materials

2.1.1 Fly ash

Electrical power generation plants that use coal as a fuel in the boilers extract large amounts of fly ash as a byproduct for the combustion process [12]. Fly ash is a fine powdered material consisting of fine particles that are driven out of the boiler by the flue gases through the chimney. ASTM C 618 classifies fly ash based on chemical requirements and physical requirements into Class F and Class C [13]. The requirements for class F is shown in **Table 1**.

Table 1. Chemical requirements [13]

	Class F
SiO ₂ +Al ₂ O ₃ +Fe ₂ O ₃ , min, %	70.0
(SO ₃), max, %	5.0
Moisture content, max, %	3.0
Loss on ignition, max, %	6.0

The fly ash used in the current study is manufactured at Iskenderun power station in compliance with DIN EN 450 – fly ash for concrete and is classified as class F in accordance to ASTM C 618. The product specifications as reported by the manufacturer is shown in **Table 2**.

Table 2. Fly ash properties

Parameter	Average
Loss On Ignition, %	2.6
Fineness > 0.045 mm, %	15.1
Chloride (Cl), %	0.012
Gross Density, kg/m ³	2330

The specific surface area of the fly ash is 0.356 gm/m², the maximum grain size is 350 µm, the mass percentage retained on 45 µm (no. 325) sieve is 64.25% which exceeds ASTM C 618 limit of 34% for fly ash designated to be used as a Pozzolan material in normal concrete. The percentage of fine materials was disregarded due to the fact that the fly ash is being used

as a cementitious material and thus, finer particles results in more chemically active material. The particle size distribution is shown in **Fig. 1**.

Fig. 1. Fly ash particle size distribution

The chemical composition analysis was conducted in the General Laboratories Department of the Iraq Geological Survey in Baghdad. The detailed results are tabulated in **Table 3**.

Table 3. Fly ash chemical composition

Oxide	SiO ₂	Al ₂ O ₃	CaO	SO ₃	LOI	Na ₂ O	K ₂ O
Content	58.13	18.89	1.60	0.18	3.28	1.27	1.46

2.1.2 Sodium Hydroxide

Commercially available sodium hydroxide (NaOH), known as caustic soda, was used for the current study. The physical appearance is white solid smooth shiny flakes. The purity of the sodium hydroxide was measured in the General Laboratories Department of the Iraq Geological Survey in Baghdad and was reported as 98.8%. The product is hygroscopic thus; it was kept in sealed airtight bags in order to protect the flakes from absorbing moisture from the air. Sodium hydroxide reacts with carbon dioxide (CO₂) from the air to form sodium bicarbonate (NaHCO₃) and this is another reason for protecting sodium hydroxide from the exposure to air.

2.1.3 Sodium silicate

Sodium Silicate (Na₂SiO₃) has been supplied from the local market. The consistency is a liquid of 0.55 water content by mass.

2.1.4 Coarse aggregate

The coarse aggregate that was used for the current study was normal weight crushed aggregate with maximum size of 12.5 mm and conforming to ASTM C 33 [14] as shown in **Fig. 2**. The bulk density of the coarse aggregate was found to be 2665 kg/m³, the absorption is 0.5% and the sulfate content is 0.08%.

Fig. 2. Course aggregate sieve analysis

2.1.5 Fine aggregate

Clean and washed river sand was used in the current study. ASTM C 33-03 specifications [14] were used for the standardization of the sand; it was found that the tested sand conformed to the specifications as shown in **Fig. 3**. The bulk density of the fine aggregate was found to be 2600 kg/m³ and the sulfate content is 0.13%.

Fig. 3. Fine aggregate sieve analysis

2.1.6 Superplasticizer

The superplasticizer used is the KUT PLAST SP 400 high range water reducing admixture of relative density 1.22-1.24 which is a solution based on modified sulfonated naphthalene formaldehyde condensate. The product was purchased from the local market and was stored in a place protected from extreme temperature.

2.1.7 Water

Clean tap water was used for the sodium hydroxide solution preparation and as an extra mixing water for more workable Geopolymer concrete.

2.2 Mixing procedure

The first step of mixing was to prepare the alkaline solution by mixing the water with the sodium hydroxide flakes with stirring by a rod for 30 seconds. The solution then was left for at least 1 hour for the complete dissolving of the NaOH flakes. Then the sodium silicate liquid was added to the NaOH solution with stirring for 1 minute and left for at least 30 minutes in the laboratory. The fine and coarse aggregate were mixed together in the electric pan mixer for 2 minutes then the fly ash was added and mixed for 1 minute. The HRWA superplasticizer was added to the alkaline solution not later than 2 minutes of the addition of the alkaline solution to the dry materials. The HRWA superplasticizer was mixed for 20 seconds with the alkaline solution, then directly added to the dry materials in the mixer. The mixing lasted for 4-7 minutes in the mixer. When extra-water was required, it was added to the wet mix at least 1 minute after the mixing of the alkaline solution with the dry materials.

2.3 Curing procedure

The curing of the Geopolymer concrete differs from that of the Portland cement concrete. The former is cured by heat, while the latter is cured by water. The specimens were cured by two methods: One of the methods is the oven discontinuous curing, which expose the Geopolymer concrete specimens to temperature in the oven for few hours then stopping the oven to let it cool down to the ambient temperature. This method was applied to mix 3 through mix 6. The curing regime is illustrated in **Fig. 4**, 5, and 6. The oven temperature is 90°C for mix 3, 4, and 5 and is 80°C for mix 6. The second curing method is exposing of the specimens to ambient temperature only.

Fig. 4. Discontinuous oven curing of mix 3

Fig. 5. Discontinuous oven curing of mix 4

Fig. 6. Discontinuous oven curing of mix 5

Fig. 7. Discontinuous oven curing of mix 6

3 Experimental study

3.1 mixture characteristics

The selection of the parameters that was based on their influence on the fresh and hardened properties of the Geopolymer concrete. Geopolymerization of the precursors of the mineral-polymer is series of chemical processes that include reactions, forming, and arranging to result in the Geopolymer structure. It has been found by many researchers that both the alkaline solution properties and the oxides in the fly ash have vast impact on the properties of the mineral-polymers [15-20]

In the current study NaOH molarity was kept constant at 12 M. The effect of the molarity of NaOH is controversial. Some studies indicated that the higher the

NaOH concentration, the higher the compressive strength [21]. Some studies show that high NaOH concentration results in lower compressive strength [17]. The molarity of the caustic soda solution affects the setting time of the Geopolymer concrete [16].

The percent of Na₂O in the solution can be controlled by the concentration of NaOH and the ratio of sodium silicates to sodium hydroxide (SS/SH), see **Fig. 8**. The fly ash used in the current study has 1.27% by weight of Na₂O. Depending on the molarity and SS/SH ratio of the mixtures shown in **Table 4**, Na₂O from fly ash can contribute in a range of (5-15)% of the total Na₂O weight of the Geopolymer.

Fig. 8. The ratio of sodium silicate to sodium hydroxide in the mixes

The weight of the fly ash in the mix is an important factor for the compressive strength of the Geopolymer concrete. The ratio of Fly ash/(Na₂O+SiO₂) and Fly ash/(Fly ash+Na₂O+SiO₂) is considered to be utilized for studying the contribution of fly ash quantity relative to Na₂O, and SiO₂ in the various mixes.

Table 4. Parameters of the Geopolymer of current study

Mix No.	Water/Fly ash weight ratio	Water/Solids weight ratio	HRWRA type	HRWRA/Fly ash (l/100 kg)	Molarity	Na ₂ SiO ₃ /NaOH weight ratio	Fly ash/(Na ₂ O+SiO ₂) (%)	Fly ash/(Fly ash+Na ₂ O+SiO ₂) (%)	SiO ₂ :Na ₂ O weight ratio (%)
1	0.35	0.30	K*	2.28	12	1.17	614	86	77
2	0.32	0.27	K*	2.57	12	2.35	665	87	113
3	0.32	0.26	K*	2.94	12	1.64	504	83	94
4	0.32	0.26	K*	3.33	12	1.66	498	83	94
5	0.32	0.26	K*	3.33	12	1.66	498	83	94
6	0.32	0.26	K*	4.12	12	1.66	498	83	94

* KUT PLAST SP 400

3.2 Mixture proportions

The mix proportions for the six mixtures of the current study is shown in **Table 5**. The quantities are for one cubic meter of Geopolymer concrete.

Table 5. Mixture proportions

Mix no.	Fly Ash (kg/m ³)	Extra water (kg/m ³)	Fine aggregate (kg/m ³)	Coarse aggregate (kg/m ³)	Sodium Silicate (kg/m ³)	NaOH solution(kg/m ³)	Superplasticizer (kg/m ³)
1	452	46	751	858	104	89	13
2	410	43	768	987	107	45	13
3	414	11	723	1000	130	79	15
4	413	9	721	998	131	79	17
5	413	9	721	998	131	79	17
6	412	9	719	995	131	79	21

The curing type, curing temperature and ambient temperature are detailed in **Table 7**

3.3 Testing Methods

3.3.1 Workability

Slump test as an indicator for the concrete workability was conducted in compliance with ASTM C 43 specifications [22]

3.3.2 Compressive strength

Cylinders with d=100 cm and h=200 cm were used to shape the Geopolymer concrete specimens. The specimens were tested using 2000 kN compression testing machine. The test specimens and procedures were conducted in compliance with ASTM C 39-05 specifications [23].

4 Results and discussion

4.1 Compressive strength

The compressive strengths of the tested cylinders are shown in **Table 7**. It can be seen that the compressive strength of mix 1 which was cured in ambient temperature of 43°C has a progressive strength with time and reached 29.21 MPa at 40 days. While for mix 2 which was also cured at ambient temperature of 43°C the compressive strength is 23.45 MPa at 40 days. Referring to **Table 5**, this increase in strength can be explained by higher percentage of the fly ash that leads to higher binder in the matrix. Another factor that led to the higher

strength for mix 1 is the Na₂SiO₃/NaOH weight ratio which when increased from 1.17 to 2.35 negatively affected the compressive strength. **Table 4** shows that water to fly ash ratio for mix 2 is 0.32 which is lower than that for mix 1 (0.35). This decrease in water was not enough to compensate the negative effect of the high SS/SH ratio. This leads to a conclusion that SS/SH ratio is a vital factor when designing Geopolymer concrete.

Mixes 3 through 6 were cured discontinuously in the oven. For mix 3, the specimens were left at ambient temperature of 42°C for the first three days after casting. Then they were exposed to oven curing for 1 hour at 90 °C then left at ambient temperature of 42°C until they were tested for the compressive strength at the fourth day (see **Fig. 4**). The development of strength was noticeable if compared to mix 1 and mix 2. One hour of discontinuous curing was enough to result in a compressive strength after only four days that is close to the compressive strength of mix 1 after 7 days and to mix 2 at 40 days. Discontinuous curing for mix 3 resulted in 40.58 MPa at 40 days age.

Discontinuous curing of 12 hours within 3 days for mix 4 (see **Fig. 5**) resulted in a compressive strength which is almost equal to that of mix 3 at 21 days. In spite of this improvement in the early strength of mix 4, the strength improvement at later ages is more obvious for mix 3 than mix 4. Both mix 3 and mix 4 were cured discontinuously for 40 days with total oven curing of 34 hours, but the compressive strength of mix 3 was 40.58 MPa which is 23% more than that of mix 4 at age 40 days. Everything is the same for mix 3 and mix 4 except the curing regime. To explain the reason behind the divergence in the compressive strength of the two mixes, it is important to mention that mix 3 was left in the ambient temperature for 3 days as a rest period prior to any oven curing. Mix 4 was directly cured in the oven after the casting in the molds. Curing regime has enhanced the geopolymerization of the goepolymer concrete through the activation of the chemical reactions of the fly ash with the alkaline solution in the early age of mix 4, but mix 3 has developed 40 days compressive strength of 23% higher than mix 4.

Mix 5 is identical to mix 4 and was left in the ambient temperature for two days of rest period then cured at 90°C for one hour then left in the ambient temperature for three days and discontinuous oven curing was then resumed. The compressive strength at 21 days of mix 5 is almost equal to that of mix 4 in spite of the fact that mix 5 oven curing time is remarkably more than that of mix 4. The effect of the rest period can be observed from the need of mix 5 for more oven curing to reach the compressive strength of mix 4 at the same age.

The influence of the rest period was also inspected in mix 6. No rest period was allowed for mix 6 (which is identical to mix 4 and mix 5). It is obvious that the progress of the compressive strength for mix 6 is higher than that for mix 5 in spite of the drop in the ambient temperature to 36°C which was 38°C in the case of mix 5. Again, this divergence in compressive strength is demonstrated by the influence of the rest period.

Table 6 compares the compressive strength of mix 3, mix 4, mix 5, and mix 6 for specific curing times and shows that for mixes without rest period, a higher early compressive strength can be obtained by the same oven curing time and in earlier age than the mixes with the rest periods.

Table 6. Early strength comparison of mixes 3 through 6

Mix no.	Rest period	Age (days)	Oven curing (hours)	Compressive strength (MPa)
3	Yes	7	12	23.60
4	No	3	12	27.00
5	Yes	8	16	24.15
6	No	7	18	31.74

5 Conclusions

One of the main expenses associated with the production of the Geopolymer concrete is the oven curing costs. The following conclusions summarize the effect of the investigated factors on both compressive strength and the cost of the Geopolymer concrete:

1. The ratio of SS/SH is an important factor that influences the compressive strength of the Geopolymer concrete and thus, trial mixes of various SS/SH for optimization are needed to result in the required compressive strength with the least oven curing. A value of 1.17 was found the best for ambient cured Geopolymer concrete.
2. Discontinuous oven curing, even for short period of 1 hour significantly improves the compressive strength. An increase of 56% in the compressive strength was achieved by one hour of oven curing.
3. In the case of discontinuous curing, the rest period when allowed to the Geopolymer concrete upon casting results in higher 40 days compressive strength than that of Geopolymer concrete which is directly oven cured. The strength improvement is remarkably evident within the period from 28 days to 40 days.
4. The early strength improvement of Geopolymer without rest period excitation is higher than when rest period is allowed. In one example, it has shown a compressive strength of 23% higher at 3 days age than the Geopolymer concrete without rest period excitation.
5. The ambient temperature plays a major role in the Geopolymerization process of the Geopolymer concrete in discontinuous curing. Within the experimental tests temperature range of (43-36)°C, the higher the ambient temperature the higher the compressive strength.

Table 7. Compressive strength of the tested cylinders

Mix no.	Curing type	Curing temperature °C	Ambient temperature °C	Age at test (day)	Hours in the oven	Compressive strength (MPa)
1	A*	43	43	7	0	22.90
				14	0	24.62
				40	0	29.21
2	A*	43	43	7	0	15.13
				14	0	16.00
				40	0	23.45
3	O**	90	42	4	1	22.00
				7	12	23.60
				18	12	25.10
				21	12	27.10
				25	12	35.77
				40	34	40.58
4	O**	90	41	3	12	27.00
				13	12	30.00
				21	12	30.78
				40	34	32.97
5	O**	90	38	8	16	24.15
				14	18	29.27
				21	55	30.35
6	O**	80	36	7	18	31.74
				11	40	32.09
				12	43	33.83

A* Cured in ambient temperature

O** Cured in an electrical oven

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