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Intermittent curing of fly ash geopolymer mortar

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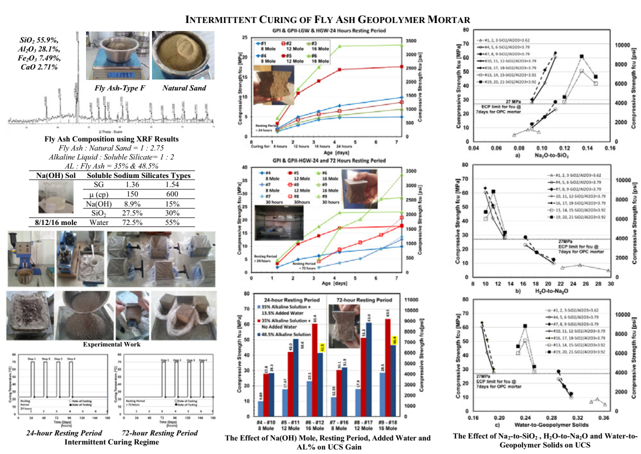
HIGHLIGHTS

- Intermittent curing at 70 °C applied on 4 steps 6 h each followed by 18 h at room temperature.
- Intermittent curing improved the UCS at the end of each curing step.
- The effect of resting period and added water content on UCS was investigated.
- Increasing the AL-to-FA content is sensitive to the Na_2O-to-SiO_2 mole ratio.
- Very low H_2O-to-Na_2O and high Na_2O-to-SiO_2 mole ratios have inverse effect on UCS.

GRAPHICAL ABSTRACT



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ABSTRACT

The research work focuses on the production of type F fly ash based geopolymer using intermittent curing. Two different types of soluble sodium silicate and Na(OH) solution with three different mole ratios were used with a fixed ratio. Two different fly ash-to-alkaline liquid activator ratios were used with and without additional water content. Two different resting periods were checked prior to starting the curing regime. The curing temperature was set at 70 °C applied intermittently on 4 steps for 6 h each per day followed by 18 h rest at ambient temperature. Twenty-one different geopolymer mixtures were cast using a mixture of fly ash and natural sand at a fixed ratio. The gain of compressive strength was checked at age 24, 48, 72, and 96 h and 7 days. Intermittent curing proved to increase the compressive strength of all geopolymer mortar at the end of each curing step. Thirteen geopolymer mixtures exceeded the Egyptian Code of Practice limit set for the 7-day compressive strength at 27 MPa. The UCS is directly proportional to the increase of the specific gravity of the soluble sodium silicate used, the age, the Na(OH) solution mole concentration, the alkaline liquid activator-to-fly ash ratio, the resting period and the Na_2O-to-SiO_2 mole ratio. Yet, it is inversely proportional to additional water content, H_2O-to-Na_2O mole ratio and the water-to-geopolymer solids ratio.

1. Introduction

Geopolymer is an inorganic polymer that results from polymerization of alumina-silicate raw materials using an alkaline activation. The alkaline liquid activator is mainly a combination of soluble sodium (or potassium) silicate and sodium (or potassium)

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hydroxide solution. The inorganic polymeric material was developed by Davidovits (1978) while geopolymer term is founded by him on 1990 [5,16]. Industrial waste materials containing a considerable amount of alumina and silica are fly ash, blast furnace slag, mine trailing, red mud... etc. Geopolymer [3,4,11,13,14] has high potential to compete with cement as a binder in the concrete technology. Cement production is one of the most highly intensive fuel consuming industries and thus highly environmentally non-friendly material. The production of 1 ton of cement clinker produces 0.5 ton of carbon di-oxide due to the calcination of limestone and 0.45 tons due to burning fuel. Geopolymer may be considered an environmentally friendly material since the production of geopolymer cement does not contribute to the greenhouse gases while it uses environmentally polluting industrial waste materials as precursors.

Geo-polymerization [3,11,13,14] process, although not fully understood, may be divided into three main phases; namely *dissolution* of Si and Al species found in the raw materials through the effect of hydroxide ions followed by *condensation* of precursor ions into monomers and finally *polymerisation* of monomers into three dimensional polymeric structures. These three steps can either take place simultaneously or concurrently with each other. Water is produced through the polymerisation process as discontinuous nono-pores in the paste. Water plays no role in the chemical reaction; it merely provides workability and initial reaction medium to the geopolymer.

Curing temperature [1–4,6–9,12,15–22] plays an important role in the exothermic reaction of geopolymer. It represents the major hurdle in the in-situ use of geopolymer in the concrete industry. High temperature acts as reaction accelerator and influences the rate of gain of strength as well as the ultimate compressive strength of the geopolymer. Different curing schemes were investigated by numerous research works. Yet, they all have common characteristics namely; heat curing time is continuous at constant maximum temperature. Elevated curing temperature and longer curing time were proved to result in higher compressive strength and high rate of gain of strength. Most literature had revealed that continuous curing for 24–48 h at a maximum temperature between 60–90 °C, can produce high strength geopolymer binder. Intermittent curing regimes were never investigated. The laboratory elevated curing utilized constant heat curing inside the oven for the full period of the curing time. Intermittent curing would have applied "a cyclic-type" of curing where high temperature is reached and maintained for a certain period of time followed by a "longer period" where temperature drops to a lesser value (ambient temperature), thus, utilizing a longer period of time. Several factors should be investigated such as molar ratios of constituents such as Na_2O-to-SiO_2, SiO_2-to-Al_2O_3, Na_2O-to-SiO_2, and H_2O-to-Na_2O, water-to-geopolymer solids ratio, and resting period. An intermittent curing scheme should be investigated to shed some light on its effect on rate of gain of strength and ultimate compressive strength of geopolymer mortar.

2. Significance of the research

This paper reports the experimental results for determining the compressive strength of geopolymer standard mortar cubes made out of fly ash type F after intermittent curing scheme at 70 °C for 4 steps for continuous 6 h followed by 18 h of rest at ambient temperature for each step. Parameters investigated include two different types of soluble sodium silicate, three different mole concentration ratios for the Na(OH) solution, one fixed ratio of Na(OH) solution-to-soluble sodium silicate, two different resting periods, two different alkaline liquid activator-to-fly ash ratios and using or omitting of additional water content.

3. The experimental programme

The experimental program, shown in Table 1, was carried out to study the effect of intermittent curing regime on the compressive strength of standard mortar cubes 70.6 × 70.6 × 70.6 mm made out of fly ash geopolymer mixtures. All fly ash geopolymer mortar mixtures were cast using a fly ash-to-natural sand ratio of 1:2.75. This ratio was chosen to mimic the same ratio used in ordinary Portland cement OPC standard mortar mixture specified by the Egyptian Code of Practice ECP [10]. The alkaline liquid activator was made out of sodium hydroxide Na(OH) of 98% purity with a Na(OH) solution-to-soluble sodium silicate liquid at a constant ratio of 1:2 by weight. Three standard mole concentrations for Na(OH) solution were used, namely; 8, 12 and 16 mol by weight. All fly ash geopolymer mortar mixtures were subjected to an intermittent curing regime of 4 steps for 6 h each at 70 °C. Eighteen resting hours separated any two successive curing steps where the electric oven was shut down leaving the temperature to drop down gradually to the ambient temperature. Twenty-one fly ash based geopolymer mortar mixtures were cast and divided into three phases.

In the first phase; two types of soluble sodium silicate were used; "low-viscous sodium silicate" LGW and "high viscous sodium silicate" HGW. The alkaline liquid activator-to-fly ash ratio was set at 35% of the fly ash content by weight. To mimic

Table 1
Geopolymer mortar mixture proportions.

#	Geopolymer mixture	Fly ash	Sand	Na(OH)	Distilled water	Glass water	Add water	Resting period	Total No. of Spec.
		g	g	g	g	g	G	hours	
1	GMI-LGW- 8M	3000	8250	85	265	700	405	24	15
2	GMI-LGW-12M	3000	8250	114	236	700	405	24	15
3	GMI-LGW-16M	3000	8250	137	213	700	405	24	15
4	GMI-HGW-8M	3000	8250	85	265	700	405	24	15
5	GMI-HGW-12M	3000	8250	114	236	700	405	24	15
6	GMI-HGW-16M	3000	8250	137	213	700	405	24	15
7	GMII-HGW-8MR	3600	9900	102	318	840	486	72	18
8	GMII-HGW-12MR	3600	9900	137	283	840	486	72	18
9	GMII-HGW-16MR	3600	9900	164	256	840	486	72	18
10	GMIII-HGW-8M-35%	600	1650	17	53	140	–	24	3
11	GMIII-HGW-12M-35%	600	1650	23	47	140	–	24	3
12	GMIII-HGW-16M-35%	600	1650	27	43	140	–	24	3
13	GMIII-HGW-8M-48.5%	600	1650	24	73	194	–	24	3
14	GMIII-HGW-12M-48.5%	600	1650	31	66	194		24	3
15	GMIII-HGW-16M-48.5%	600	1650	38	59	194	–	24	3
16	GMIII-HGW-8MR-35%	600	1650	17	53	140	–	72	3
17	GMIII-HGW-12MR-35%	600	1650	23	47	140	–	72	3
18	GMIII-HGW-16MR-35%	600	1650	27	43	140	–	72	3
19	GMIII-HGW-8MR-48.5%	600	1650	24	73	194	–	72	3
20	GMIII-HGW-12MR-48.5%	600	1650	31	66	194		72	3
21	GMIII-HGW-16MR-48.5%	600	1650	38	59	194	–	72	3

Conversion factor: divide the weight in (g) by 453.6 to get weight in (lb).