Performance of Geopolymer Concrete Exposed to Freezing and Thawing Cycles

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Abstract- *In this study, the effect of rapid freezing and thawing (ASTM C666 – procedure A) on three different types of Geopolymer concrete studied using three types of pozzolanic material: fly ash, metakaolin and ground granulated blast furnace slag (GGBFS). The Geopolymer concrete was prepared using 400 kg of the pozzolanic material with alkaline liquid prepared at 8 molar concentration with normal fine and coarse aggregates. The ratio of alkaline to fly ash and GGBFS was 1.5: 1 and for metakaolin was 2: 1 for workability and compressive strength requirements. Specimens (100 × 100 × 400) mm were exposed to 100, 200 and 300 cycles of freezing and thawing. The decrease in measured compressive strength was (23, 43, and 26%) for Fly ash, metakaolin and GGBFS respectively. The investigated types of concrete showed good resistance to freezing and thawing. The durability factor of these types was (77%, 68%, and 81%) for fly ash, metakaolin, and GGBFS respectively.*

Keywords- *Freezing and thawing, Fly ash, Geopolymer, GGBFS, Metakaolin.*

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1. Introduction

The freezing starts with the large spaces and then moves to the smaller spaces [1]. Concrete, which is dry or not exposed to water, not damaged by the freezing and thawing, British Cement Association (BCA) in 1997 showed the ratio of the occurrence of the damage of freezing and thawing to the concrete structures as shown in Table 1 [2].

As known when water freezes its volume increase up to 9%, the concrete exposed to low temperatures (frost) water in the pores will begin frozen gradually that causes pressure on the non-freezing water because of the expansion of ice volume, this pressure causes internal tensile stresses lead to damage in future [3].

The appropriate method for determining the resistance of concrete to freezing and thawing is by examining the freezing and thawing according to ASTM C666, which includes two procedures [4]:

- Procedure A, in which the specimens surrounded by water, i.e. the process of freezing and thawing is in water.
- Procedure B the specimens surrounded by air in the phase of freezing, but the thawing is in water.

Skvara et al. [5] use method B to test the Alkali Activated Fly ash (AAF) mortar, where it presented to 150 cycles. Compressive strength reduced to 70% compared to non-exposed specimens.

Table 1: Types of concrete structures and their exposure to Freezing and Thawing [2]

Type of structure	(%) incidence of freezing and thawing cause of damage
Bridges	6
Buildings	4
Hydraulic structures: Massive Small	17 20
Marine	10
Car parks	17

Geopolymer paste (cylindrical specimens 27.6*50 mm) made from thermally treated kaolin and fluidized bed combustion bottom ash (FBC-BA) as part or whole kaolin replacement and specimens processed at laboratory temperature (22ºC). Slavik et al. [6] exposed these specimens to 50 cycles of freezing (for 2 hours -20°C)/thaw (for two hours in water) according to the European standard EN 14617-5:2005. It found that compressive resistance decreased to 80%.

Steinerova [7] studied the effect of freezing and thawing on Metakaolin based Geopolymer mortar using quartz sand, where cured at laboratory temperature and pressure in sealed plastic foils for 3 weeks. Specimens of dimensions 40 × 40 × 160 mm subjected to 25 cycles of freezing and thawing, the decrease in compression resistance measured by the percentage of sand used. Which means the following: If the specimens contain enough Geopolymer matrix binder to avoid coarse pores, the frost-resistance trend rises with

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the sand content, up to 82 wt.%. Above this limit, the excessive sand caused coarse pores to appear, leading to a decrease in frost resistance.

Temuujin et al. [8] used two types of fly ash class C with calcium oxide ratio of 14% and 30% to production Geopolymer concrete. The specimens subjected to 40 cycles of freezing and thawing, the mixtures containing alkali solution containing only (8M) NaOH not affected, while the mixtures containing alkali solution consisting of (10M) 50% sodium hydroxide + 50% sodium silicate did not withstand more than 5 cycles.

Henrik et al. [9] make a comparison of ordinary cement mortar with Geopolymer mortar made from slag and fly ash with different percentages of alkali solution with quartz sand (0-2) mm and a percentage of glass fiber. The specimens cured at laboratory temperature. After 28 cycles of freezing and thawing, the relative dynamic modulus elasticity of Geopolymer mix was 96% compared to the conventional cement mixture 91%.

In this study, the effect of freezing and thawing on three types of Geopolymer concrete made from different type of pozzolanic materials (fly ash, Metakaolin, and GGBFS), exposed to 300 cycles and according to ASTM C666 procedure A, which is considered the most serious. The loss of weight studied and the measurement of compression and flexural resistance. After every 100 cycles, the durability factor (DF), the static and dynamic modulus of elasticity calculated by ultrasound examination.

2. Experimental Work

The experimental program included the preparation of the raw materials for the manufacture of the Geopolymer concrete and tests, design of the mixtures for each type of Geopolymer concrete, preparation of specimens, exposed to freezing and thawing, at last the laboratory tests, and compare the results. Figure 1 showed the experimental program.

I. Source Materials

Three types of source materials were used, they are: Fly ash, Metakaolin, and GGBS. Turkish hard coal fly ash from Iskenderun power station was used, Local kaolin clay was burnt at 700ºC for one hour to allow it to change into Metakaolin. GGBS is the by-product of iron, which collected from BASF Company. XRF results for these materials are shown in Table 2 from Iraqi Geological Survey.

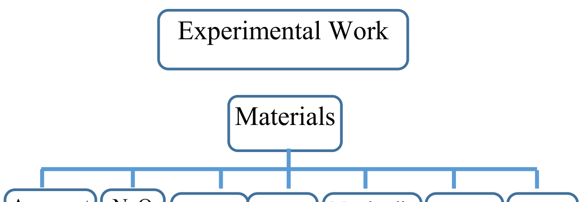

Figure 1: Flow chart of Experimental work

Table 2: Chemical composition and surface area of used source materials

Oxides %	Fly ash	Metakaolin	GGBS
SiO_2	63.0	56.77	30.7
Al_2O_3	27.1	30.85	13.3
Fe_2O_3	4.12	2.48	0.35
CaO	1.20	0.58	42.4
MgO	0.74	0.59	6.89
Others	3.71	8.73	6.32
Specific surface area (m^2/kg)	778	17250	681

II. Aggregate

Crushed gravel with (12.5mm) maximum size used, as coarse aggregate, natural graded sand was the fine aggregate according to ASTM C33[10], as shown in Tables 3 and 4. The fineness modulus of fine aggregate is (2.66).

III. Alkaline Solution

Alkaline liquid obtained by blending sodium silicate and sodium hydroxide solutions. Industrial type sodium silicate with chemical composition of Na_2O = 13.5%, SiO_2 = 32.5%, H_2O = 54%. Sodium hydroxide NaOH flakes with 97-98% purity used. The sodium hydroxide solution was prepared by dissolving the NaOH flakes in tap water with different concentration as required.

IV. High-Range Water Reducer

A high range water reducer superplasticizer (KUT PLAST SP400) based on modified sulfonated naphthalene formaldehyde condensate was used to enhance workability of Geopolymer concrete.

1. Mix Design

Mixes implemented for each type of source materials, as shown in Table 5.

Table 3: Sieve analysis of coarse aggregate and materials less than 75micron

sieve size (mm)	Passing (%)	Requirements gradation
19	100	100
12.5	98.5	90-100
9.5	63.9	40-70
4.75	2.7	0 – 15
2.36	0.2	0 – 5
0.075	0.66	1 % upper limit
SO_3%	0.048	0.1%Upper limit

Table 4: Sieve analysis of fine aggregate and materials less than 75micron

sieve size (mm)	Passing (%)	Requirements gradation
9.5	100	100
4.75	98.2	95 – 100
2.36	90.8	80 – 100
1.18	73.3	50 – 85
0.6	52.3	25 – 60
0.3	15.1	5 – 30
0.15	4.2	0 – 10
0.075	1.98	3% upper limit
SO_3%	0.387	0.5% upper limit

Table 5: Mix proportions of Geopolymer concrete

Materials (kg/m^3)	Fly Ash(FA)	Metakaolin (MK)	GGBFS (GG)
Mass	400	400	400
NaOH (8M)	19	26	26
Sodium Silicate	103	200	150
Water	54	73	73
Fine aggregate	650	650	650
Coarse aggregate	1200	1200	1200
HRWR	12	18	18

2. Preparation of Test Specimens

After casting the Geopolymer concrete in molds (100*100*400) mm^3, the molds placed in the oven for 24 hours. Then the specimens taken out and removed from their molds. After that, they additionally cured in the oven for another 48 hours. Fly ash based Geopolymer [11], GGBS based Geopolymer cured in 65±5 ºC [12], and the Metakaolin based Geopolymer cured in 45±5 ºC [13]. Then the specimens taken out and allowed to cure at room temperature until 28 days, then put all the specimens in three containers full with water and put the containers in the Climatic controlled cabinet (freezing and thawing chamber) to exposed to 100, 200, and 300 cycles according to ASTM C666 Procedure A.

3. Results and Discussion

Twelve prism specimens of each type of mixes, six specimens were put into the freezing and thawing chamber, 2 specimens examined after every 100 cycles. Another six specimens were stored simply in the controlled room until the age of test.

I. Weight Change

The results of this study show that the Metakaolin based Geopolymer concrete is the most type that weight change due to freezing- thawing cycles, as shown in Figure 2. This may be due to the higher absorption rate, which was higher than the other two types as shown in Table 6. The absorption rate indicates the increase of pores in the matrix; the effect of freezing- thawing cycles is the generation of pressure inside the pores due to the increase in the size of the frozen water and thus increase the size of pores. Although no using of air entrainment additives, the Geopolymer concrete of all types showed excellent performance in weight change. There are no any deformation or even cracking due to exposure to 300 cycles of freezing and thawing in the most serious case, samples were immersed in water throughout the test period as shown in Figure 3.

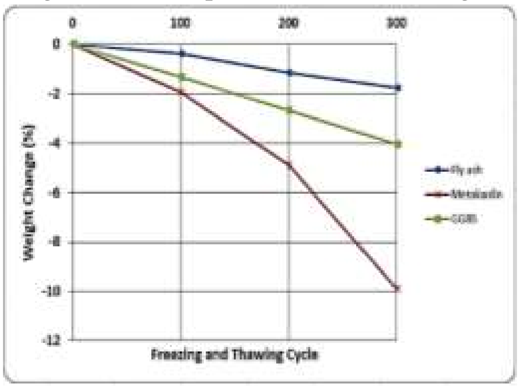

Figure 2: Effect freezing and thawing cycles on weight change of different type geopolymer concrete

Table 6: Density, weight change, and absorption rate for types of Geopolymer concrete

Mix Type	No.of cycles	Density (kg/m3)	Weight change (%)	Absorption rate (%)
FA	0	2287	0	1.83
	100	2279	-0.35	
	200	2261	-1.14	
	300	2247	-1.75	
MK	0	2237	0	2.71
	100	2194	-1.92	
	200	2128	-4.87	
	300	2016	-9.88	
GG	0	2330	0	2.13
	100	2299	-1.33	
	200	2268	-2.66	
	300	2236	-4.03	

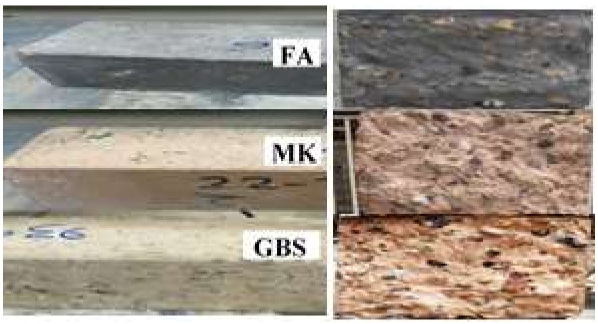

Figure 3: Different type of Geopolymer concrete specimens after 300 cycles of freezing and thawing

II. Ultrasonic pulse velocity test

The speed of the waves measured according to ASTM C597-02 [14]for the specimens of the Geopolymer before and after exposure to the freezing and thawing test using the Ultrasonic apparatus. The velocity of the waves (V) in any concrete depends on the elasticity and density of concrete. The pulse velocity (V) calculated by dividing L by T, where (L) is the distance between the transmitter and the receiver in (m), (T) the time in (sec.).

The speed of the waves depends on the density of the sample, so it was noticed that waves speed decreased with the increase in the number of cycle of freezing and thawing due to voids within the sample as shown the results in Table 7. After calculate the waves velocity (V), dynamic modulus of elasticity (Ed) found by the Eq. (1) [14]

$$E = \rho V^2 \frac{(1-2\mu)(1+\mu)}{(1-\mu)} \qquad (1)$$

Where (E) is the modulus of elasticity, (ρ) is the density of Geopolymer concrete, (μ) is Poisson's ratio, and V pulse velocity. The Poisson's ration of Geopolymer concrete ranged (0.16 – 0.19), so the term $\frac{(1-2\mu)(1+\mu)}{(1-\mu)}$ ranged between (0.91 – 0.94), so taken as (0.92) in all calculations.

III. Flexural and compressive strength

The test was performed in accordance with ASTM C78-09 [15] for specimens exposed to freezing and thawing and non-exposed specimens. Noted from Table 8 that flexural resistance decreases with increasing exposure cycles as shown in Figure 4. Compressive strength in the equivalent cube method was performed on the remaining parts of the prism samples after completion of the flexural test for the Geopolymer concrete, which also observed decreasing compressive strength continuously with increasing cycles of freezing and thawing as show in and Figure 5.

Table 7: The pulse velocity and dynamic modulus of elasticity results for types of Geopolymer concrete

Type of mix	No.of cycles	Pulse velocity (m/sec)	E_d (GPa)
FA	0	4100	35.37
	100	4098	35.21
	200	3976	32.88
	300	3349	23.19
MK	0	3802	29.75
	100	3490	24.59
	200	3421	22.91
	300	2776	14.29
GG	0	4016	34.57
	100	3897	32.12
	200	3738	29.15
	300	3444	24.40

Table 8: Flexural and compressive strength for types of Geopolymer concrete types

7	No.of cycles	Flexural strength (MPa)	Reduction in flexural strength (%)	Compressive strength (MPa) [equivalent cube]	Reduction in compressive strength (%)
FA	0	6.38	0	58.82	0
	100	5.05	20.8	55.79	5.1
	200	4.65	27.1	49.45	15.9
	300	4.42	30.7	45.05	23.4
MK	0	3.35	0	25.45	0
	100	2.21	34.0	21.52	15.4
	200	1.95	41.8	19.56	23.1
	300	1.85	44.8	16.73	34.3
GBS	0	3.46	0	49.46	0
	100	3.01	13.0	44.74	9.5
	200	2.42	30.0	38.05	23.1
	300	2.29	33.8	36.51	26.2

When comparing the results for flexural strength and compressive strength after 300 cycles of freezing thawing with non-exposed samples, the decrease in flexure strength of the Geopolymer concrete specimens were (31, 45 and 35%) for ash fly ash, Metakaolin and GGBFS based Geopolymer concrete respectively. The decrease in compressive strength after 300 cycles of fly ash, Metakaolin and GGBFS based geopolymer concrete were (23.4, 34.3 and 26.2)%, respectively. Noted that the decrease in flexure strength is higher than the decrease in compressive strength. This is due to the effect of the freezing- thawing cycles that cause microscopic cracks in the matrix. These microscopic cracks have the effect of reducing the flexural strength rather than compressive strength.

The decrease in porosity in the geopolymer concrete improves its resistance to freezing and thawing. The results obtained indicate that decrease in porosity led to reduce weight change and increases flexure strength or compressive strength.

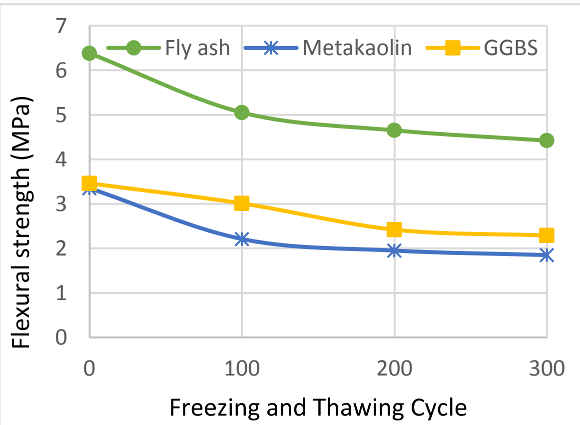

Figure 4: Effect Freezing and thawing cycles on flexural strength of Geopolymer concrete

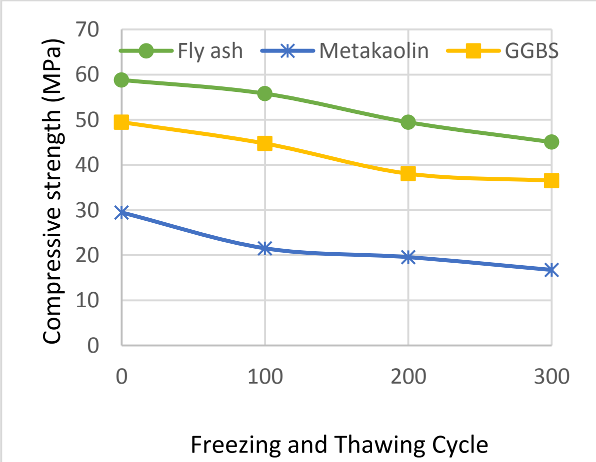

Figure 5: Effect Freezing and thawing cycles on compressive strength of Geopolymer concrete

IV. Durability Factor

Depending on ASTM C666-97 procedure A, where freezing and thawing in water, which considered the most harmful condition because of water ingress into pores at the time of melting and freezes again lead to increase in size of pores, calculated the relative dynamic modulus of elasticity and durability factor from the equations below:

Pc = (Edn1/Edn)*100% (2)

Where:

Pc: relative dynamic modulus of elasticity, after c cycles of freezing and thawing, percent,

Edn: dynamic modulus of elasticity at 0 cycles of freezing and thawing, and

Edn1: dynamic modulus of elasticity after c cycles of freezing and thawing.

DF = P*N/M (3)

Where:

DF: durability factor of the test specimen,

P: relative dynamic modulus of elasticity at N cycles, %,

N: number of cycles at which P reaches the specified minimum value for discontinuing the test or the specified number of cycles at which the exposure is to be terminated, whichever is less, and

M: specified number of cycles at which the exposure is to be terminated.

Results showed that the three types of Geopolymer concrete showed good resistance even after 300 cycles where relative dynamic modulus of elasticity did not reach less than 60% of the initial modulus of elasticity (except Metakaolin at 300 cycle), so the durability factor in fact equal to the relative dynamic modulus of elasticity because (N=M). The results shown in Table (9) and the Figures (6,7) showed the fly ash and GGBS based Geopolymer concrete have more resistance than Metakaolin based Geopolymer concrete, that due to its high strength and low porosity.

Table 9: Relative dynamic modulus of elasticity of Geopolymer concrete

Type of mix	No. of cycles	Relative Dynamic Modulus of Elasticity (% of E_d @0 cycle)	Durability factor
FA	0	100	100
	100	99.5	99.5
	200	93.0	93.0
	300	65.6	65.6
MK	0	100	100
	100	82.6	82.6
	200	77.0	77.0
	300	48.0	45.9
GG	0	100	100
	100	92.9	92.9
	200	84.3	84.3
	300	70.6	70.6

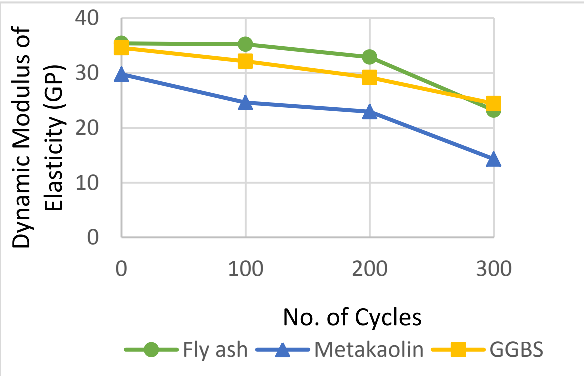

Figure 6: Effect freezing and thawing cycles on Dynamic modulus of elasticity of Geopolymer concrete

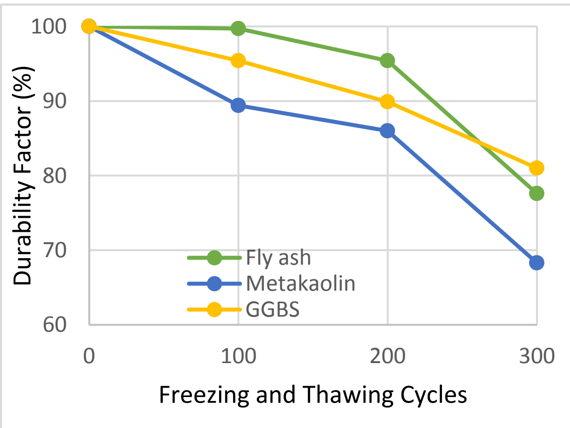

Figure 7: Durability Factor for all types of Geopolymer concrete with freezing and thawing cycles

6. Conclusion

- The percentage of weight loss directly proportional to the ratio of Geopolymer concrete absorption of water.
- The Ultrasonic test results give an indication of the density and homogeneity of the Geopolymer concrete, i.e. the increase of the Geopolymerization process results, which lead to increased density and reduced voids.
- The voids in the Geopolymer concrete due to the freezing and thawing cycles lead to a decrease in compressive and flexural resistance.
- The Durability factor (DF) is equal with relative dynamic modulus of Elasticity because the last did not reach less than 60% of the initial modulus of elasticity for all Geopolymer concrete types (except Metakaolin at 300 cycle).
- The fly ash and GGBFS based Geopolymer concrete were more resistant to freezing and thawing test than the Metakaolin based Geopolymer concrete, less weight loss and high compression resistance due to the less porosity resulting from the homogenization of the Geopolymarization process due to the increased amorphous silica and alumina in fly ash and high calcium oxide in GGBFS.

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