Effect of Curing System on Metakaolin Based Geopolymer Concrete

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Abstract

Geopolymer concrete is considered as a new material that brought attention from many researchers due to its low impact on environment pollution compared to normal Portland cement concrete. This research studied the effect of different curing systems on the strength of Metakaolin (as silica-alumina material) based Geopolymer. Eleven curing systems were used including curing by sun light and laboratory ambient environment at winter (with temperature of 8-19^{o}C) and at summer (with temperature of 32-48^{o}C), curing with halogen lamp, curing by heat at 60^{o}C for 6 hours and at 100^{o}C for 4 hours, water curing, curing by wet burlap, in addition to a mixture of different previous curing systems.

The results indicated that the optimum curing temperature for Geopolymer concrete is (32-48^{o}C)that can be accomplished under sun light or room temperature, while moist curing proved to be not suitable for this type of concrete. The results also indicate that this type of concrete has high rate of hardening, where it is possible to gain more than 83% of the 28 days strength at 7 days age when using optimum temperature curing.

Keywords: Metakaolin, Sun light, Halogen lamp, Alkaline solution, Compressive strength.

الخلاصة

تعتبر الخرسانة الجيوبوليمرية من المواد الانشائية الحديثة التي جذبت اهتمام الكثير من الباحثين لما لانتاجها من تأثير قليل على تلوث البيئة مقارنة مع الخرسانة الاعتيادية الحاوية على السمنت البورتلاندي. تم في هذا البحث دراسة تاثير نوع المعالجة على مقاومة الخرسانة الجيوبوليمرية المعتمدة على الميتاكاؤلين كمادة سليكية-الومينية، حيث اعتمدت احدى عشرة طريقة شملت المعالجة بحرارة الشمس وجو المختبر في فصلي الشتاء (بدرجة حرارة ٨-١٩ م) والصيف (بدرجة حرارة ٣٢-٤٨ م)، المعالجة بمصباح الهالوجين، المعالجة بالحرارة بدرجة ٦٠ م لمدة ٦ ساعات ومئة درجة مئوية لمدة ٤ ساعات، المعالجة بالماء،المعالجة بتغطية النماذج بالجنفاص المرطب بشكل مستمر، اضافة الى خليط من طرق المعالجة السابقة.

بينت النتائج ان افضل درجة حرارة معالجة للخرسانة الجيوبوليمرية هي (٣٢-٤٨م) والتي يمكن ان تتم في ضوء الشمس او في جو الغرفة، بينما وجد بان المعالجة الرطبة غير مناسبة لهذا النوع من الخرسانة. كما بينت النتائج بان معدل التصلب عالي لهذه الخرسانة، حيث يمكن الحصول على اكثر من ٨٣% من مقاومتها بعمر ٢٨ يوم عند عمر ٧ ايام.

الكلمات المفتاحية: الميتاكاؤلين، ضوء الشمس، مصباح الهالوجين، المحلول القلوي، مقاومة الانضغاط.

Introduction

Geopolymer gets more interest because it produces low CO_2-emission in comparison with Portland cement. Geopolymer shows similar or better engineering properties compared to cement (Wallah and Rangan, 2006)**.** Portland cement production increases annually by 3% with the increasing of construction industries. This environmental pollution by CO_2 emission in addition to consuming huge amount of virgin materials by cement manufacture led the researchers to look for alternative binders that are more sustainable and economical (Satpute *et al.,*2012).

The reaction of the materials having aluminosilicate with others having high alkalis (hydroxide and/or silicate solution) produces a synthetic alkali aluminosilicate material generically called a 'Geopolymer'(Davidovits, 2011).Today Geopolymer concrete (based on Metakaolin or Fly Ash) became widespread in construction industry sector and considered appropriate alternative that has been used in many applications including precast beams, boat ramp, pavement, bricks, retaining wall, water tank, precast bridge decks…etc. (Aldred and Day, 2012).

The polymerisation process involves a substantially fast chemical reaction of aluminosilicate minerals under alkaline condition that results in a three dimensional polymeric chain. Three basic steps lead the chemical reaction: 1. dissolution of Si and Al atoms from the source material, 2. Orientation or condensation of precursor ions into monomers, and 3. setting or polycondensation of monomers into polymeric structures (Duxson *et al.,*2007). The final products of Geopolymerisation are influenced by many factors regarding chemical composition of the source materials and alkaline activators (Diaz *et al.,* 2010; Duxson *et al.,* 2007; Yip *et al.,* 2008).

The polymerisation process is generally accelerated by temperature more than 35°C (Davidovits, 2011). The acceleration of Geopolymerisation process depend on the raw material that having aluminosilicate. Fly Ash based Geopolymer produced in ambient temperature achieve lower strength in the early days as compared to heat cured samples (Vijai *et al.,*2010), while Metakaolin needs less temperature for curing.

The Geopolymer concrete as a relatively new construction material, still needs to explore. Hence this work aims to study the effects of using different curing systems on the produced Metakaolin based Geopolymer concrete.

Materials

Kaolin clay collected from Dewekhla region, Al-Anbar Governorate. The kaolin grinded and then burned in furnace up to 700ºC ± 20ºC, for 1 hour to produce the Metakaolin, according to (Ibrahem and Wahab, 2008). After that the Metakaolin grinded to specific surface area of 23 m^2/g. The chemical compositions of Metakaolin that adequate to the requirements of Pozzolan ASTM C618 are shown in Table (1).

The aluminosilicate binder (Metakaolin) were activated by a mixture of sodium hydroxide and sodium silicate solutions. Sodium hydroxide is commercially available in the pellet form. Sodium hydroxide solution with desired concentration (10 Molar) was prepared by mixing 98% pure pellets with tap water. The used sodium silicate solution (manufactured in UAE) has a ratio of SiO_2 to Na_2O of 2.4, which is 32.5 percent SiO_2, 13.4 percent Na_2O and 54.1 percent of water.

A high-range water reducer, SP, was used for the production of rheoplastic concrete. It is based on a sulphonated naphthalene polymer.

The fine aggregates (sand) were obtained from Al-Ukhadir region, Karbala Governorate. The grading and sulfate content were conforming to the requirements of Iraqi specification IQS No.45/ 1984–Zone 2.The coarse aggregate was crushed gravel with maximum size of 19 mm. The grading of this aggregate conforms to the Iraqi specification IQS No.45/ 1984, (5-19 mm). Physical properties and size distribution of aggregates are presented in Table (2).

Table 1: Chemical composition for used Metakaolin

Oxide composition	Oxide content %
SiO_2	51.59
Al_2O_3	38.11
Fe_2O_3	1.82
CaO	0.45
MgO	0.23
SO_3	0.14
K_2O	0.43
Na_2O	0.11
L.O.I	6.12

Table 2: Properties of aggregates

Properties	Coarse aggregate			Fine aggregate		
	Sieve size (mm)	Percent passing	Limits of Iraqi Standard IQS 45-1984	Sieve size (mm)	Percent Passing	Limits of Iraqi Standard IQS 45-1984, zone 2
Sieve analysis	20	100	100	10	100	100
	14	95.5	90-100	4.75	96	90-100
	10	75.8	50-85	2.36	78	75-100
	5	6.5	0-10	1.18	64	55-90
	2.36	/	----	0.6	53	35-59
				0.3	26	8-30
				0.15	6	0-10
Specific gravity	2.65		----	2.6		----
Absorption	0.9%		----	1.8%		----
Fine material	0.09		1.0 percent maximum	1.3		3.0 percent maximum
Sulfate content	0.069		0.1 percent maximum	0.12		0.5 percent maximum

Mix design of Geopolymer concrete

In the design of Geopolymer concrete mix, coarse and fine aggregates together were taken as 75% of entire mixture by mass. This value is similar to that used in OPC concrete in which it will be in the range of 75 to 80% of the entire mixture by mass. Fine aggregate was taken as 40% of the total aggregates (720 kg/m^3 of sand while the coarse aggregate was 1100 kg/m^3). The Metakaolin binder content was kept constant as 400 kg/m^3.

The alkaline activator was prepared in the laboratory by mixing the sodium hydroxide solution with sodium silicate solution for 24 hours before actual concrete mixing to enhance reactivity of the solution. The percent of sodium hydroxide to the sodium silicate was 1: 3.5 while the percent of alkaline solution was 45% of Metakaolin weight. 12 kg/m^3 of sulphonated naphthalene superplasticizer added to water (which is 10% of Metakaolin weight) then added to the alkaline solution and mix together for about 2 min.

The Metakaolin, fine and coarse aggregates were mixed by pan mixer in dry condition for three minutes and then the alkaline solution was added (with superplasticizer and extra water) and mix for another four minutes to prepare the Geopolymer concrete (Hardjito *et al.,* 2004). The Geopolymer concrete was casted in 100 mm cube molds and compacted by a vibrating table. After 24 hours, the specimens were demolded.

Curing System for Geopolymer concrete

Different types of curing studied in this research are to find out the optimum way for curing Metakaolin Geopolymer concrete. Some of curing system tested in different seasons to study the effect of variation in temperature between summer and winter especially in Iraq. Compressive strength considers a scale for selection the best type for curing that compatible with Metakaolin Geopolymer concrete. Table 3 illustrates the types of curing systems which were applied to the Geopolymer concrete samples after demolding them till testing age. Each results consists of test three cubes, where the total number of specimens was 78 cube.

Results and Discussion

Table (3) shows the effect of different curing systems on Metakaolin Geopolymer concrete compressive strength.

1-Sun light curing:

Sun light curing in summer gave the highest compressive strength at 7 days when the temperature was (36-48)°C, while the compressive strength decreased clearly in winter when the temperature became (12-19)°C. Temperature accelerates the formation of hard structure especially in the early stage of Geopolymerisation reaction (Rovnaník 2010). The increasing in compressive strength between summer and winter was 33.61% at 7 days and 29.57% at 28 days**.**

2- Laboratory curing:

Compressive strength for laboratory curing was too close to sun light curing, as in the case in of sun light curing, Geopolymer concrete with laboratory curing has high compressive strength at high temperature (in summer) while there is a reduction in strength when temperature decrease (in winter). The temperature (32-37)°C considered good device for gaining strength and helping the Geopolymerisation that needs temperature more than 35°C (Davdovits, 2011). The compressive strength was higher by 36.84 and 30.66% at 7 and 28 days age respectively in summer compared with that at winter**.**

3- Halogen curing:

The halogen curing system conducted by using (lamp 1000 watt). The halogen lamp provides heat reach to (50-55)°C, this heat is important to accelerate Geopolymerisation process. The compressive strength of this type of concrete was lower than those using summer sun light curing and laboratory curing by about 14%, that refer to by increasing temperature the compressive strength values slightly decrease which can indicate that higher curing temperature makes the hardened structure less dense, and hence less compact (Rovnaník, 2010).

Halogen curing very important especially in winter when the temperature decrease, because of providing appropriate temperature that contribute to activate Geopolymerisation process.

4- Water curing:

Water curing for Metakaolin Geopolymer concrete consider ineffective for providing Geopolymer concrete with moderate compressive strength. This type of curing was unsuitable and cause a reduction in strength, maybe because the water entering inside concrete cause volume change leading to cracks in the structure of Geopolymer (Kirschner and Harmuth, 2004).

5- Wet burlap curing:

Moisture curing is conducted by using continuously wetting burlap with water to keep the specimen moist. The samples kept in temperature ranging between (20±4)°C. For the same causes of water curing, moisture curing consider unfit to produce Geopolymer concrete with suitable strength. The condensation polymerization that takes place is endothermic in nature therefore supply of heat is required (Nuruddin *et al.,* 2011).

6- Heat cycle I and laboratory curing:

Heat cycle I and laboratory curing started after removing samples from the molds in two stages. First stage included heat curing at 60°C for 6 hours in the oven then the second stage started involving keeping the specimen in the laboratory till the day of testing. As compare with laboratory curing alone at summer when temperature between (32-37)°C compressive strength lowering by using heat curing at 60°C by

28.77% at 7 days and 24.55% at 28 days. This lowering in strength as a result of increasing temperature to 60°C lead to accelerate Geopolymerisation process and affected adversely on the structure of Geopolymer by increasing voids and lowering density (Rovnaník, 2010).

7- Heat cycle I and sun light curing:

Heat cycle I and sun light curing including first step heat curing at 60°C for 6 hours in the oven, second step involved keeping the samples under sun light at temperature (36-48)°C. As compare with sun light curing in summer, the reduction in compressive strength by using heat curing reach to 25.51% at 7 days and 22.98% at 28 days. This lowering in strength as a result of increasing temperature to 60°C lead to accelerate Geopolymerisation process and affected adversely on the structure of Geopolymer by increasing voids and decreasing density (Rovnaník, 2010).

8- Sun light and Laboratory curing:

Sun light and laboratory curing, this type of curing including combination between sun light curing in summer with temperature (36–48)°C for first 7 days then laboratory curing with temperature (32– 37)°C to the day of testing in 28 days. In this type of curing the compressive strength reached to the maximum value at 28 days. The appropriate temperature at first 7 days accelerate the Geopolymerisation process then the other 21 days with moderate temperature lead to continuous Geopolymerisation with homogenous structure and less porosity that effect positively on compressive strength (Davidovits, 2011).

9- Halogen and Laboratory curing:

The combination of two types of curing starting with halogen curing with 1000 watt lamp for 7 days then laboratory curing at (32-37)°C continues to 28 days (day of test). As compare with previous system (Sun light and Laboratory curing), this type of curing system has slight reduction in compressive strength. The reduction in strength result to increasing temperature at earlier ages by using halogen system when the temperature was between (50-55)°C leading to increase the Geopolymer-isation degree therefore, the amount of reaction products increases. On the other hand, the increasing in strength combined by low density and inhomogeneous structure therefore, at longer ages there is reduction in compressive strength. The explanation of this behavior is similar to the influence of temperature on the strength development of Portland cement (Rovnaník, 2010).

10- Heat cycle II and sun light curing:

Heat cycle II and sun light curing started with heat curing in the oven at 100°C for 4 hours then sun light curing begging in the summer with temperature (48–36)°C to 7 days and 28 days. As compare with sun light curing only, compressive strength decrease for this type by 46.26% at 7 days and 47.89% at 28 days.

11- Heat cycle II and halogen curing**:**

Heat cycle II and halogen curing system this combination started with heat curing at 100°C for 4 hours in the oven then halogen lamp (500 watt) curing started with temperature between (50–55)°C. For comparison with halogen curing only, heat curing at 100°C affect adversely on compressive strength by 44.08% at 7 days and 44.76% at 28 days.

Generally, high temperature of curing Metakaolin Geopolymer concrete reduces the compressive strength. Researchers have different explanation for the effect of high temperature of curing. First one, by increasing temperature more than 60°C the compressive strength reduce because of changing phase from amorphous phase to crystalline phase. The crystalline phase having high porosity and voids and that lead to lowering compressive strength by increasing temperature. Curing temperature affects the generation rate of crystalline phase by increasing temperature, the process of changing Geopolymerisation gel (amorphous phase) to crystalline phase more rapidly. On the other hand when curing Metakaolin Geopolymer concrete at ambient temperature there is no trace to crystalline phase (Tosheva and Valtchev, 2005).

The second opinion believe that increasing temperature of curing of Metakaolin Geopolymer concrete, lead to losing the water of mixture and that cause voids in the structure of Geopolymer concrete and lowering compressive strength (Rovnaník, 2010).

Increasing the temperature of curing leads to increase of Geopolymerisation process therefore there is increasing in compressive strength at earlier ages but at later ages there is reduction in strength. At low temperature 10°C Geopolymerisation process is very slow therefore compressive strength very low in spite of only amorphous phase available, that refers to low temperature not effective for Metakaolin Geopolymer concrete.

Sun light curing system and laboratory curing in summer consider a suitable system for curing Metakaolin Geopolymer concrete. Such a case is very convenient and economical in hot weather countries such as Iraq. In cold weather it is recommended to adopt another system of curing such as curing with halogen light. Generally the optimum temperature for curing Metakaolin was between (48 – 32) °C depending on this research and that matching with (Davidovits, 2011). Figure 1 illustrates the compressive strength of Metakaolin Geopolymer concrete at 7 days curing in different system. Also, Figure 2 shows compressive strength at 28 days age.

Table 3: Results of curing systems on Metakaolin Geopolymer concrete strength

Mix No.	Curing system		Compressive strength, MPa, at age:	
			7 days	28 days
1	Sunlight	Summer T (36-48) °C	28. 53	30.63
2		Winter T (12-19) °C	18.94	21.57
3	Laboratory	Summer T (32-37) °C	28.25	29.97
4		Winter T (8-15) °C	17.84	20.78
5	Halogen 1000 Watt lamp, the temperature ranging (50-55) °C		23.68	26.74
6	Water		13.11	14.87
7	Wet burlap		16.52	18.03
8	Heat cycle I and laboratory curing (heat curing 60°C for 6 hr. and laboratory curing in when T (32-37) °C		20.12	22.61
9	Heat cycle I and Sunlight curing (heat curing 60°C for 6 hr. and Sunlight curing when T (36-48) °C		21.25	23.59
10	Sunlight and Laboratory curing in Summer		-	31.19
11	Halogen and Laboratory curing In Summer		-	27.10
12	Heat cycle II and Sunlight (heat curing 100°C for 4 hr. and Sunlight curing when T°C (36-48)		15.33	15.96
13	Heat cycle II and Halogen (heat curing 100°C for 4 hr. and Halogen by 1000 Watt lamp)		13.24	14.77

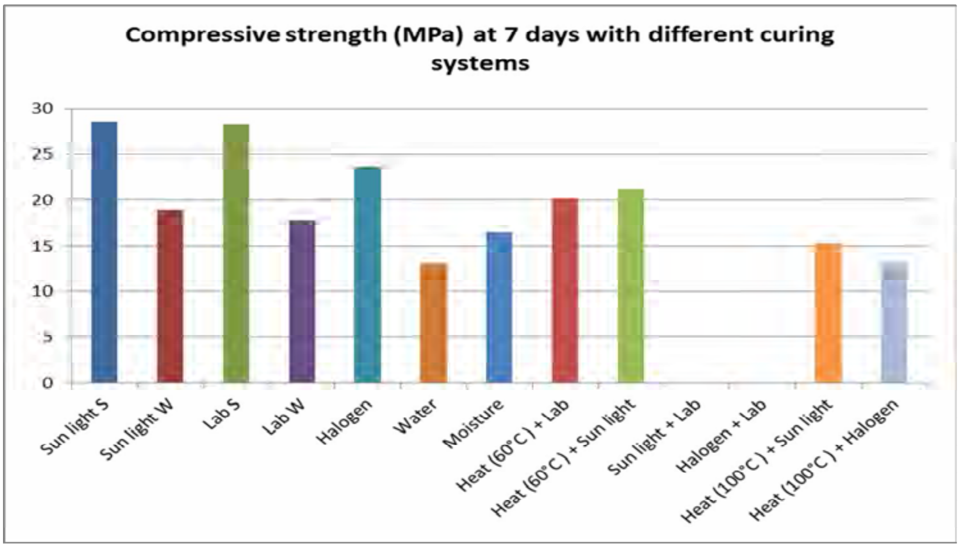

Figure 1: Compressive strength at 7 days for different curing systems.

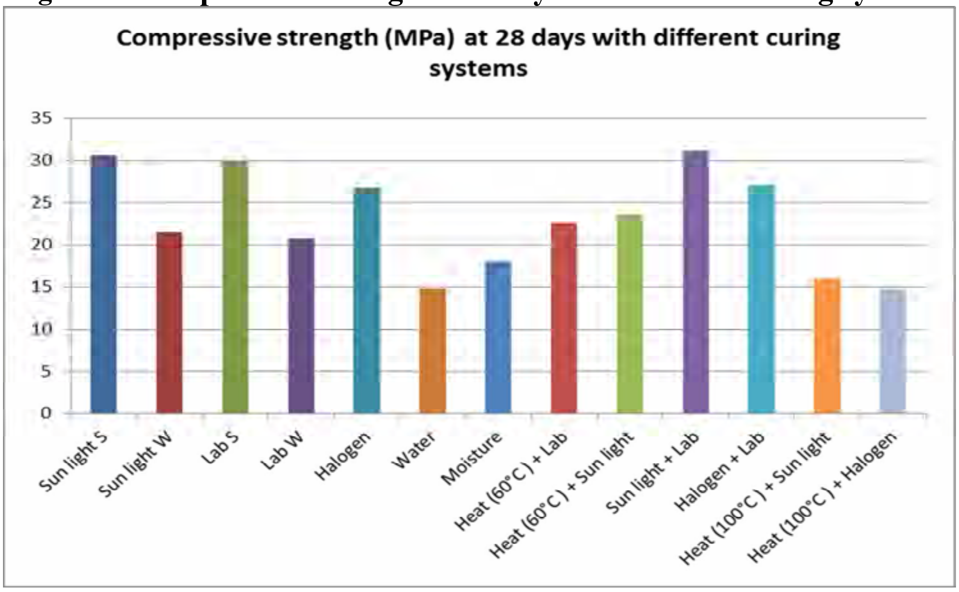

Figure 2: Compressive strength at 28 days for different curing systems.

Conclusions

Following conclusions were drawn from the experimental results of this study by varying curing system and curing temperature.

1. The temperature is very important factor effect on Geopolymerisation process, the optimum temperature for curing Metakaolin Geopolymer concrete is (32–48)°C under sun light or room temperature.
2. Metakaolin Geopolymer concrete can gain more than 93% of 28 days at only 7 days age when using optimum temperature curing.
3. Sun light curing and laboratory curing in the summer consider effective and cheap device for curing Metakaolin Geopolymer concrete when the temperature more than 35°C.
4. Halogen curing system is provide appropriate temperature (50-55)°C for curing Metakaolin Geopolymer concrete in cold weather.
5. Wet burlap and water curing are not a suitable system for curing the Geopolymer concrete.

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