

# Compressive Strength and Interfacial Transition Zone Characteristic of Geopolymer Concrete with Different Cast In-Situ Curing Conditions

Muhd Fadhil Nuruddin, Andri Kusbiantoro, Sobia Qazi, Nasir Shafiq

**Abstract**—The compressive strength development through polymerization process of alkaline solution and fly ash blended with Microwave Incinerated Rice Husk Ash (MIRHA) is described in this paper. Three curing conditions, which are hot gunny curing, ambient curing, and external humidity curing are investigated to obtain the suitable curing condition for cast in situ provision. Fly ash was blended with MIRHA at 3%, 5%, and 7% to identify the effect of blended mixes to the compressive strength and microstructure properties of geopolymer concrete. Compressive strength results indicated an improvement in the strength development with external humidity curing concrete samples compared to hot gunny curing and ambient curing. Blended mixes also presented better performance than control mixes. Improvement of interfacial transition zone (ITZ) and micro structure in external humidity concrete samples were also identified compared to hot gunny and ambient curing.

**Keywords**—Compressive Strength, alkaline solution, fly ash, geopolymer, ITZ, MIRHA,

## I. INTRODUCTION

GEOPOLYMER concrete, named after the reaction between polymer and geological origin source material, was proposed to replace all cement portions in concrete as the main binder [1]. The reduction of cement portion in concrete is expected to decrease the Portland cement demand; hence reducing the environmental issues generated from cement production.

Geopolymer concrete is commonly produced from alkaline liquid and source material. The alkaline liquid is a combination of sodium hydroxide or potassium hydroxide with sodium silicate or potassium silicate [2]. The utilization of single alkaline hydroxide activator will have lower rate reaction compared to those containing soluble silicate [3], therefore sodium silicate solution is added to sodium hydroxide solution to enhance the reaction rate between alkaline liquid and source material [4].

Combinations of fly ash and microwave incinerated rice husk ash (MIRHA) were used as source materials in this research. These low calcium materials were chosen because

high calcium content in source material can affect the polymerization process [5]. Meanwhile blended composition of fly ash and MIRHA was intended to understand the effect of different silicate content in source material since MIRHA has higher silicate content than fly ash [6].

Numerous researches have been conducted to produce quality geopolymer concrete that is comparable to OPC concrete. However the curing method has generated some limitations to the geopolymer concrete applications. The heat requirement in the curing process is supplied by electrical instruments hence it is only suitable to be used in precast concrete industry. The workability issue also needs to be addressed since some alkaline liquid and source material are not compatible with certain commercial admixture. Therefore this research is focused on the curing method to obtain geopolymer concrete that has better workability in fresh state, which is suitable for cast in situ application.

## II. EXPERIMENTAL METHOD

### A. Materials

The material selection in this research was based on specific requirement. 8 molar NaOH supplied was 99% purity and in pellet form. While  $\text{Na}_2\text{SiO}_3$  was obtained with proportion of  $\text{Na}_2\text{O}$ : 14.73%,  $\text{SiO}_2$ : 29.75%, and water: 55.52%.

Fly ash used in this research with chemical compositions as shown in Table I. Table I also shows the chemical composition of MIRHA. The rice husk used to produce MIRHA was first dried under direct sunlight to remove its moisture content hence preventing excess smoke generated from combustion process. Rice husk was then incinerated in microwave incinerator with temperature setting 400°C. The UTP Microwave Incinerator (UTPMI) used in the research adopted the Air Cooled Magnetron system with an overall dimension of 2.3(H)x4.0(W)x4.0(L) with a chamber capacity of 1 m<sup>3</sup>. MIRHA was then ground in ball mill for 2000 times to increase its fineness. Coarse aggregates were prepared under saturated surface dry (SSD) condition with maximum aggregate size of 20 mm. Since commercial water reducing admixture was not suitable for mixtures in this research, sucrose solution was utilized in the mixture to delay the

Muhd Fadhil Nuruddin, Andri Kusbiantoro, Sobia Qazi, and Nasir Shafiq are with Civil Engineering Department, Universiti Teknologi PETRONAS, Bandar Seri Iskandar, 31750 Perak, Malaysia, phone: +60-5-3677282; e-mail: fadhilnuruddin@petronas.com.my

setting time during mixing and casting process.

### B. Experimental Setup

This research was designed with several mixture proportions incorporating blended source material to investigate their effect on geopolymer concrete performance. The mixture proportions are shown in Table II. NaOH and Na<sub>2</sub>SiO<sub>3</sub> were prepared 1 hour before mixing process to prevent precipitation of NaOH in the solution.

TABLE I  
FLY ASH AND MIRHA CHEMICAL COMPOSITIONS [6],[7]

Oxide	MIRHA	Fly Ash (%)
SiO <sub>2</sub>	88.90 %	51.19 %
Al <sub>2</sub> O <sub>3</sub>	0.16 %	24.00 %
Fe <sub>2</sub> O <sub>3</sub>	0.45 %	6.60 %
CaO	0.63 %	5.57 %
MgO	0.72 %	2.40 %
SO <sub>3</sub>	0.32 %	0.88 %
K <sub>2</sub> O	3.65 %	1.14 %
Na <sub>2</sub> O	-	2.12 %

TABLE II  
MIX PROPORTIONS EMPLOYED

Mix Code	Fly Ash (kg/m <sup>3</sup> )	MIRHA (kg/m <sup>3</sup> )	CA (kg/m <sup>3</sup> )	FA (kg/m <sup>3</sup> )	NaOH (kg/m <sup>3</sup> )	NaSiO <sub>2</sub> (kg/m <sup>3</sup> )	Water (kg/m <sup>3</sup> )	Sugar (kg/m <sup>3</sup> )
A1	350	0	1200	645	41	103	35	10.5
A2	339.5	10.5	1200	645	41	103	35	10.5
A3	332.5	17.5	1200	645	41	103	35	10.5
A4	325.5	24.5	1200	645	41	103	35	10.5
B1	350	0	1200	645	41	103	35	10.5
B2	339.5	10.5	1200	645	41	103	35	10.5
B3	332.5	17.5	1200	645	41	103	35	10.5
B4	325.5	24.5	1200	645	41	103	35	10.5
C1	350	0	1200	645	41	103	35	10.5
C2	339.5	10.5	1200	645	41	103	35	10.5
C3	332.5	17.5	1200	645	41	103	35	10.5
C4	325.5	24.5	1200	645	41	103	35	10.5

Mixing process was divided into two stages, dry mix and wet mix. Initially, coarse aggregate, fine aggregate, fly ash (and MIRHA) were mixed together in rotating pan mixer for 2.5 minutes. Alkaline and sucrose solutions were then poured into the dry mixed material and continued for wet mixing for 1.5 minutes. Fresh geopolymer concrete was then hand mixed to ensure the mixture homogeneity.

Fresh concrete was then cast in 100mm cube moulds and compacted using poker vibrator. Curing process in this research is divided into three curing methods; hot gunny curing (33-38°C), ambient curing (27-32°C), and external exposure curing (39-44°C).

In hot gunny curing, concrete samples were covered with hot gunny sack for 48 hours, with hot gunny replaced every 24 hours. To prevent heat being released immediately, the samples were covered with plastic sheet. In ambient curing, concrete samples were placed outside the room but protected from direct sunlight and rain. In external exposure curing, concrete samples were placed in a transparent chamber that was placed outside the building. The heat radiation from sunlight penetrated into the chamber but the samples were protected from rain water.

Hardened concrete samples were tested for their compressive strength at 3, 7, 28, and 56 days for all curing regime. Analysis on ITZ and microstructure properties was conducted using scanning electron microscopy (SEM) on 56 days concrete sample.

### III. RESULT AND DISCUSSION

#### A. Compressive Strength Test

The results obtained from compressive strength test illustrate polymeric process in geopolymer concrete with various curing treatment.

Fig. 1 demonstrated the compressive strength development of hot gunny curing samples. The presence of high humidity from hot gunny could not improve concrete performance, indeed it decreased the concrete strength significantly because the heat resulted from hot gunny could not be maintained for a long duration.

The condensation polymerization that takes place is endothermic in nature therefore supply of heat need to be consistently present for at least 48 hours. This process is different from OPC concrete as geopolymer concrete does not utilize water in its polymeric reaction. This in turn helps the mixing and casting process to increase the fresh concrete workability.

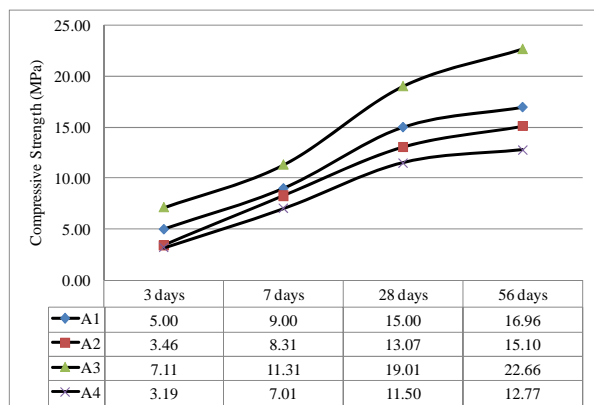


Fig. 1 Compressive strength development of concrete sample with hot gunny curing

During the maturing process, the water was expelled and evaporated from hardened concrete sample. Spaces that were previously occupied by water existed as micropores inside concrete. These pores provided a microcrack path and led to the premature failure of concrete when exposed to compressive load, hence resulting in low compressive strength performance. However, with mixture of 95% fly ash and 5% MIRHA increased concrete strength up to 36%. It indicated that the modification of Si:Al ratio in source material affected the concrete performance.

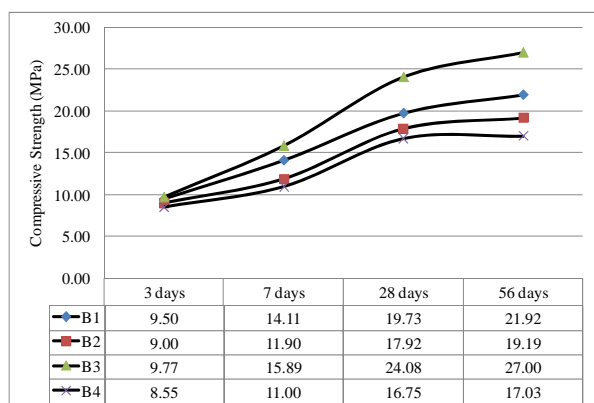


Fig. 2. Compressive strength development of concrete sample with ambient curing

In ambient curing, mixture of 95% fly ash and 5% MIRHA also performed better performance than other ambient curing samples as illustrated in Fig. 2. This mixture improved the geopolymer concrete strength up to 24% compared to non blended mixture in the same curing regime.

With higher amount of heat involved in external exposure curing, the geopolymer concrete increased its compressive strength significantly as shown in Fig. 3.

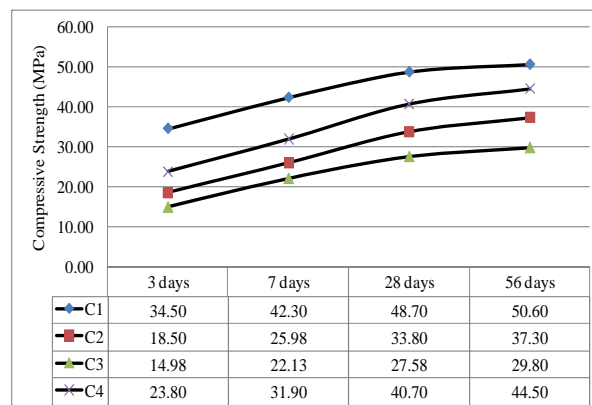


Fig. 3. Compressive Strength Development of Concrete Sample with External Exposure Curing

Different from previous curing methods, non blended samples in external exposure curing had higher compressive strength than blended samples. It indicates that modification of Si:Al ratio in source material has significant effect in humid environment but little effect in elevated temperature environment. However, non blended samples in external exposure curing had compressive strength of 147% and 92% higher compared to hot gunny and ambient curing respectively. It was also observed that the critical period of geopolymer concrete strength development is within the first week from mixing process. Lack of polymeric reaction during this period reduced the compressive strength.

#### B. Scanning Electron Microscopy (SEM) Analysis

SEM analysis was carried out to observe the microstructure properties of geopolymer concrete mainly on its ITZ. Fig 4 shows the ITZ in hot gunny curing concrete sample. This gap decreased the concrete strength by providing path to the microcracks that made them interconnected. The presence of the gap between aggregate and paste was due to the insufficient polymeric reaction during maturing.

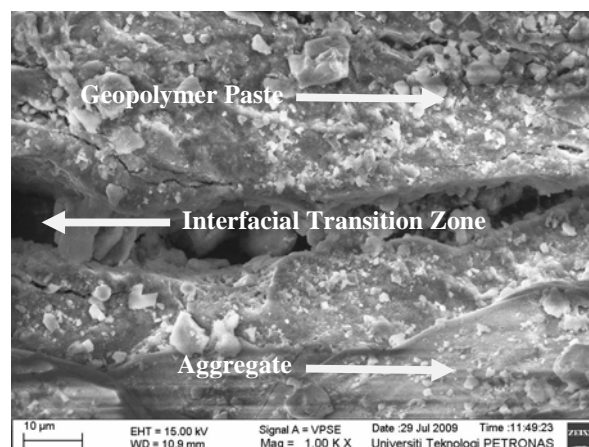


Fig. 4. SEM Image of Hot Gunny Curing Sample

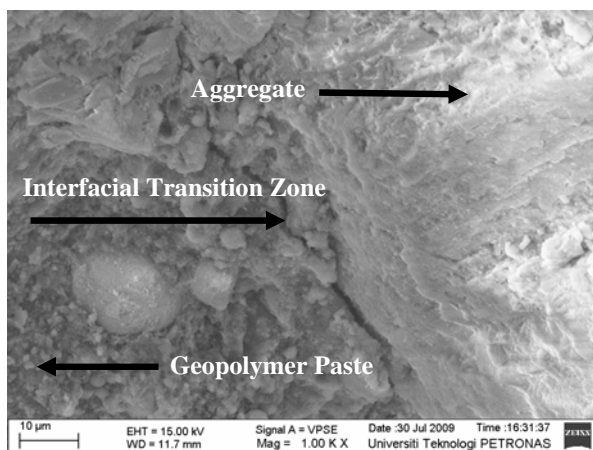


Fig. 5. SEM Image of Ambient Curing Sample

Concrete sample under ambient curing showed small ITZ width than hot gunny curing as presented in Fig 5. Solid paste and micropores distribution in concrete determined the concrete performance; therefore smaller ITZ width prevented the concrete from premature failure. It explained that the compressive strength performance of ambient curing samples had higher result than hot gunny curing.

The refinement of ITZ was evident in external exposure samples. In Fig 6 the microcrack was discontinued when entering the ITZ, hence resulting in higher compressive strength compared to the other curing method. It supported the analysis that elevated temperature was required to accelerate the polymeric reaction during early maturing period.

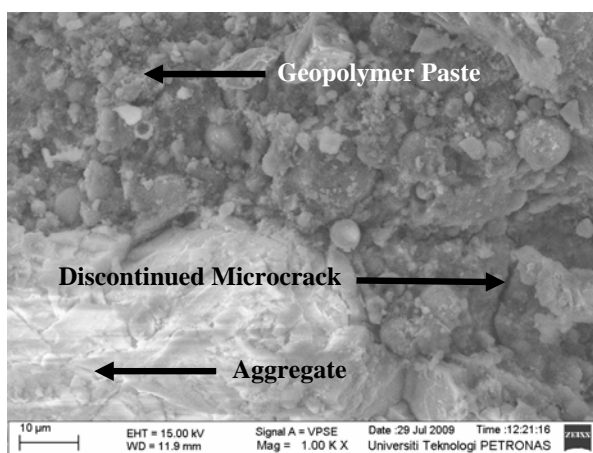


Fig. 6. SEM Image of External Exposure Curing Sample

#### IV. CONCLUSIONS

The compressive strength development of geopolymer concrete was much affected by the curing condition during maturing period. Therefore proper curing method was important to obtain acceptable geopolymer concrete structures. The external exposure curing condition used in this research was an acceptable technique to produce good concrete structures. This led to a conclusion that cast in-situ application (Malaysian climate or equivalent) was a viable

alternative and geopolymer concrete is not necessarily limited only to precast industry.

There was also a significant improvement if blended source material was used. Mixture of 95% fly ash and 5% MIRHA generated compressive strength 36% and 24% higher than non blended mixture in hot gunny curing and ambient curing respectively. While external exposure curing method generated compressive strength 147% and 92% higher than concrete sample in hot gunny and ambient curing respectively.

Improvement of ITZ was also achieved by geopolymer concrete sample in external temperature curing condition. Disconnected microcrack path led to a higher capability of concrete in sustaining heavier load.

#### ACKNOWLEDGMENT

The authors would like to thank Universiti Teknologi PETRONAS, Malaysia for the financial support in undertaking the research work.

#### REFERENCES

- [1] Davidovits, J. "High Alkali Cements for 21st Century Concretes." Concrete Technology: Past, Present and Future. P. K. Mehta, ACI, Detroit, USA. SP 144-19: 383-397, 1994.
- [2] Barbosa, V.F.F., K. J. D. MacKenzie, C. Thaumaturgo. "Synthesis and Characterisation of Materials Based on Inorganic Polymers of Alumina and Silica: Sodium Polysialate Polymers." International Journal of Inorganic Materials 2(4): 309-317, 2000.
- [3] Palomo, A., M. W. Grutzeck, M.T. Blanco. "Alkali-Activated Fly Ashes, A Cement for the Future." Cement and Concrete Research 29(8): 1323-1329, 1999.
- [4] Xu, H. and J.S.J. van Deventer. "The Geopolymerisation of Alumino-Silicate Minerals." International Journal of Mineral Processing 59(3): 247-266, 2000.
- [5] Gourley, J. T. Geopolymers; Opportunities for Environmentally Friendly Construction Materials. Materials 2003 Conference: Adaptive Materials for a Modern Society, Sydney, Institute of Materials Engineering Australia, 2003.
- [6] MF. Nuruddin, Kusbiantoro, A, Shafiq, N. "Microwave Incinerated Rice Husk Ash (MIRHA) and its Effect on Concrete Properties", IMS International Conference, American University of Sharjah, 2008.
- [7] Chin, S.C. "Performance of Used Engine Oil in Fresh & Hardened States of Normal and Blended Cement Concretes." MSc Thesis, University Teknologi PETRONAS, 2007.