Development of Geopolymer Concrete with Different Curing Conditions

M.F. Nuruddin[1], A. Kusbiantoro[1], S. Qazi[1], M.S. Darmawan[2], and N.A. Husin[2]

***Abstract*—Environmental issues resulted from cement production have become a major concern today. To develop a sustainable future it is encouraged to limit the use of this construction material that can affect the environment. Cement replacement material was proposed to partially replace cement portion in concrete. Geopolymer is a part of inorganic polymer material that has similar bonding function like cement in concrete. It consists of alkaline solutions and geological source material. Alkaline liquids used in this research are 8 M sodium hydroxide (NaOH) solution and sodium silicate (Na_2SiO_3) solutions, while source materials are fly ash and microwave incinerated rice husk ash (MIRHA). Three different curing regimes, namely hot gunny curing, ambient curing, and external exposure curing, were applied to obtain suitable method that was suitable with cast in situ application. Geopolymer concrete samples were tested on their compressive strength and microstructure properties. It was found that external exposure curing had the highest compressive strength compared to other two curing methods. Scanning electron microscopy analysis also showed better improvement in interfacial transition zone for concrete sample with external exposure curing.**

Keywords—**geopolymer, sodium hydroxide, sodium silicate, fly ash, MIRHA**

***Abstrak*—*Dampak terhadap lingkungan akibat produksi semen telah menjadi masalah yang besar pada saat ini. Untuk mengembangkan masa yang akan datang yang lebih berkelanjutan maka diperlukan usaha untuk membatasi penggunaan material konstruksi ini yang dapat mempengaruhi lingkungan. Material pengganti semen telah diusulkan untuk mengganti sebagian porsi semen dalam beton. Geopolimer adalah bagian dari polimer bukan organik yang mempunyai sifat mengikat seperti semen pada beton. Material tersebut terdiri dari cairan alkalin dan material dari sumber geologi. Cairan alkalin yang dipakai dalam penelitian ini adalah cairan 8 M Natrium Hidrosikda (NaOH) dan cairan Natrium silikat (Na_2SiO_3), sementara material sumber geologi adalah Abu Terbang dan Abu Sekam Padi yang dibakar memakai gelombang-mikro (microwave incinerated rice husk ash/MIRHA). Tiga macam cara perawatan, yaitu perawatan memakai karung panas, perawatan suhu ruang dan perawatan di tempat terbuka tanpa perlindungan dari cahaya matahari, telah digunakan untuk mendapatkan metoda yang tepat untuk aplikasi cor setempat. Contoh beton geopolimer telah diuji kuat tekannya dan sifat-sifat struktur mikronya. Dari hasil uji tersebut ditemukan bahwa perawatan di tempat terbuka tanpa perlindungan dari cahaya matahari mempunyai kuat tekan tertinggi dibandingkan dengan dua cara perawatan lainnya. Analisa dengan cara pemindaian memakai mikrosokop elektron juga menunjukkan perbaikan zone transisi antar muka (interfacial transition zone) untuk beton dengan perawatan di tempat terbuka tanpa perlindungan dari cahaya matahari.*

Kata Kunci—***geopolimer, sodium hydroksida, sodium silikat, abu terbang, MIRHA***

I. INTRODUCTION

In 1978, J. Davidovits initiated inorganic polymeric material that can be used to react with another source material to form a binder. The application of this binder is recently being focused to replace Ordinary Portland Cement (OPC) portion in concrete [1]. The environmental issues resulted from OPC production has taken the progress of polymer researches further nowadays. Depletion of raw material and CO_2 emission resulted from fuel combustion and decomposition of limestone has put cement industry as one of the main source of environment pollutants [2].

The encouragement to produce environment friendly concrete can be achieved by limiting the utilization of raw material, decreasing pollutant rate from respective OPC production, and diminishing the cement portion in concrete [3]. Employment of waste material like fly ash, rice husk ash, and other cement replacement material (CRM) can only replace cement portion until certain percentage. Geopolymer, named after the reaction between polymer and geological origin source material, is proposed to replace all cement portions in concrete as the main binder [3-4].

The main constituents of geopolymer are alkaline liquid and source material. Alkaline liquid is usually a combination of sodium hydroxide or potassium hydroxide with sodium silicate or potassium silicate [5]. The use of only alkaline hydroxide activator will result in low rate reaction compared to those containing soluble silicate [6]. The addition of sodium silicate solution to sodium hydroxide solution will enhance the reaction rate between alkaline liquid and source material [7].

Source materials used in this research are combination of fly ash and Microwave Incinerated Rice Husk Ash (MIRHA). Both of these materials have similar specification for calcium content, which is low in calcium. High calcium content in source material is not recommended since it can obstruct the polymerization process [8]. Blended composition of fly ash and MIRHA is intended to observe the effect of different silicate content in source material since MIRHA has higher silicate content than fly ash [2].

Experimental set up on geopolymer concrete has been conducted by several researchers. However the curing method has developed into some limitations to the geopolymer concrete applications. Heat requirement in

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the curing process can only be supplied by electrical instruments; hence geopolymer concrete currently can only be applied in precast concrete industry. Therefore this research is focused in the curing method to obtain geopolymer concrete that suitable with cast in situ application.

II. METHOD

This research was done by specifying the material and set up experimentally.

A. Materials

Alkaline liquids in this research were in pellet form with 99% purity. 8 Molar NaOH solutions were used for all mix proportions in this research. While Na_2SiO_3 was with proportion of Na_2O: 14.73%, SiO_2: 29.75%, and water: 55.52%.

Fly Ash used in this research consist oxide compositions as described in Table 1. 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^3. MIRHA was then ground in ball mill for 2000 times to increase its fineness. The oxide content of MIRHA is described in Table 2. Coarse aggregates were prepared under saturated surface dry (SSD) condition with maximum 20 mm in size. Since commercial water reducing admixture was not suitable for mixtures in this research, sucrose solution was utilized in the mixture to delay the setting time during mixing and casting process.

B. Experimental Setup

Mixture proportion was designed with different amount of blended source material to investigate their effect to geopolymer concrete properties. Constant amount of NaOH and Na_2SiO_3 were used throughout the mix proportions. The optimum ratio between Na_2SiO_3 and NaOH was adopted from the research done by Hardjito and Rangan in 2005. Detail for each mixture is described in Table 3. Alkaline solutions were prepared 1 hour before mixing process started to prevent precipitation of NaOH in the solution. 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 100 mm cube moulds and compacted using poker vibrator. Curing process in this research is divided into three curing method; hot gunny curing, ambient curing, and external exposure curing. In hot gunny curing, concrete samples were covered with hot gunny sack for 48 hours, with hot gunny was 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 while still protected from direct sunlight and rain. In external exposure curing, concrete samples were placed in a transparent chamber that placed outside, hence heat radiation from sunlight can penetrate into the chamber while still protect the samples from rainfall.

Hardened concrete samples were tested for their compressive strength on 3, 7, 28, and 56 days for all curing regime. Analysis on Interfacial Transition Zone (ITZ) and microstructure properties was conducted using Scanning Electron Microscopy (SEM) on 56 days concrete sample.

III. RESULTS AND DISCUSSION

A. Compressive Strength Test

The results obtained from compressive strength test illustrate polymeric process in geopolymer concrete with various curing treatment. Compressive strength development of geopolymer concrete for all curing regime is described in Table 4.

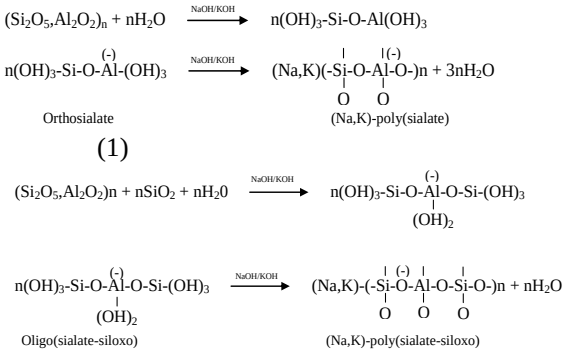
In hot gunny curing, compressive strength did not develop rapidly. The presence of high moisture content from hot wet gunny decreased the concrete strength significantly because the heat resulted from hot gunny could not be maintained for a long duration. In contrast with conventional OPC concrete, water presence in geopolymer concrete did not take part in polymeric reaction. It helps only during mixing and casting process to increase the workability.

However, during drying period (after 48 hours), water will evaporate from hardened concrete and leaved micropores inside the concrete. The presence of these pores will lead to a premature failure of concrete sample. Blended source material with 95% fly ash and 5% MIRHA provided better result than other samples in hot gunny curing as showed in Figure 1. The compressive strength for blended sample could increase until 36% compared to non-blended sample in the same curing regime It indicates that different amount of Al-Si material in fly ash and MIRHA affecting the properties of geopolymer concrete.

In ambient curing, heat generated from the environment was absorbed by the polymeric material to initiate the reaction. In this regime, compressive strength development also performed similar trend with hot gunny curing. However, the difference of heat amount in first week of maturing period has affected the performance of these two methods. Concrete sample in ambient curing did not have excess water in their environment from first day; hence polymeric reaction could take place faster than those in hot gunny curing. Blended specimen could bestow the compressive strength up to 24% compared to non-blended sample in the same curing regime and 64% compared to non-blended hot gunny concrete sample. Similar with hot gunny mixture, 95% fly ash and 5% MIRHA performed better performance than other ambient curing samples as described in Figure 2.

Meanwhile different characteristic was performed by external exposure curing. Sufficient amount of heat during daylight has accelerated the polymeric reaction inside concrete samples. Figure 3 shows that non blended sample provided the best development among all concrete samples. In average, concrete sample in external exposure curing has compressive strength 162% higher if compared to hot gunny curing sample, and

102% higher compared to ambient curing sample. It was also observed that the critical period for geopolymer concrete is within the first week from mixing-casting process. Lack of polymeric reaction within this period will result in lower performance of geopolymer concrete. Alike conventional OPC concrete, strength development in geopolymer concrete was also started to stable after 28 days. The chemical reaction for three different curing methods is the same and follow equation 1 and 2 [4, 9], except that the amount of polymer/monomer is higher as the condensation polymerization proceeds.



Higher strength development obtained in external exposure curing was due to an increase in polarization of OH^- to break Si-O and Al-O bonds on fly ash surface [10]. Condensation of these monomers to dimmer, trimmers and other low molecular elements will result in polymeric covalent bonding, i.e. poly(sialate), poly (sialate-siloxo), poly (sialate-disiloxo), etc [9, 11].

B. Field Emission Scanning Electron Microscopy (FESEM) Analysis

FESEM analysis was carried out to observe the microstructure properties of geopolymer concrete. Figure 4, 5, and 6 shows the inner condition for hot gunny curing, ambient curing, and external exposure curing concrete samples respectively.

Figure 4 shows the presence of micro crack at interfacial transition zone between aggregate and paste. Micro crack in here is a part of interfacial transition zone in the geopolymer concrete. In the freshly mixed concrete, water films occurred around the aggregate, most likely in the large aggregate particles. This higher water-binder ratio at the larger aggregate surface was responsible in the formation of porous framework than that in the mortar matrix. It is believed that this weak framework was removed during the cutting and trimming process of the FESEM specimen hence revealed the former water-filled voids at ITZ. It indicates low bonding strength of geopolymer paste caused by few amount of heats absorbed during polymeric reaction in maturing period. This weak ITZ will be connected to another micro cracks and micro pores path within paste matrix when concrete sample loaded with compressive force, which could result in premature failure of concrete sample.

In Figure 5, the presence of ITZ was covered by paste matrix hence resulted in higher compressive strength of concrete samples. Even though it did not completely cover the paste matrix, the bonding strength gained could give better performance than hot gunny curing.

Superior strength performance of external exposure curing sample was verified by the FESEM image in Figure 6. It supports the analysis that elevated temperature is important to accelerate the polymeric reaction in geopolymer concrete. With strong bonding between aggregate and paste, microcrack path will be discontinued when certain loading applied on the concrete and resulting in higher compressive strength of concrete sample.

NOMENCLATURE

In order to develop quality geopolymer concrete proper curing method is a necessity. In environment with high moisture content, blended source material with 95% fly ash and 5% MIRHA could improve geopolymer concrete performance up to 24% compared to non-blended concrete. However in elevated temperature, blended source material did not significantly improve concrete strength compared to non-blended concrete. But still the concrete compressive strength in external exposure curing could reach 162% higher than hot gunny curing and 102% higher than ambient curing. It indicates that sufficient heat amount is needed to increase polymeric reaction rate.

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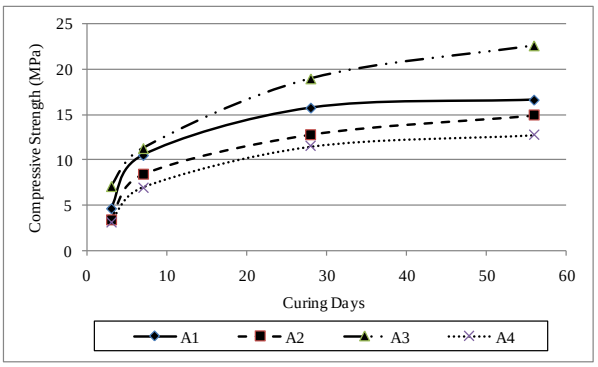

Figure 1. Compressive strength development of concrete sample with hot gunny curing

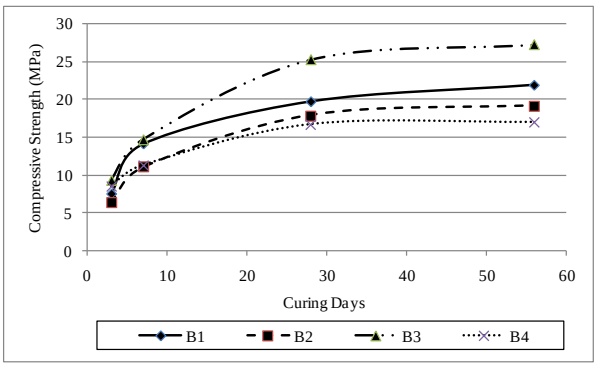

Figure 2. Compressive strength development of concrete sample with ambient curing

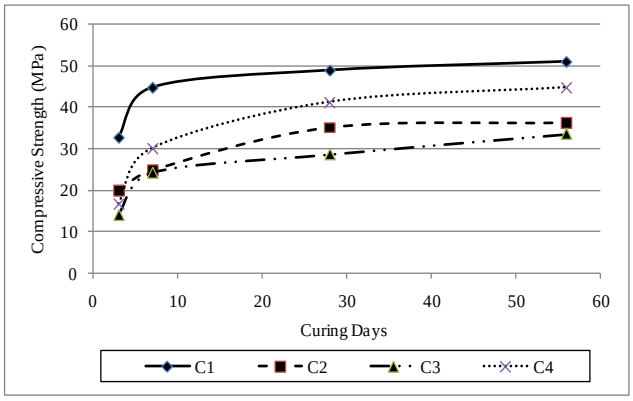

Figure 3. Compressive strength development of concrete sample with external exposure curing

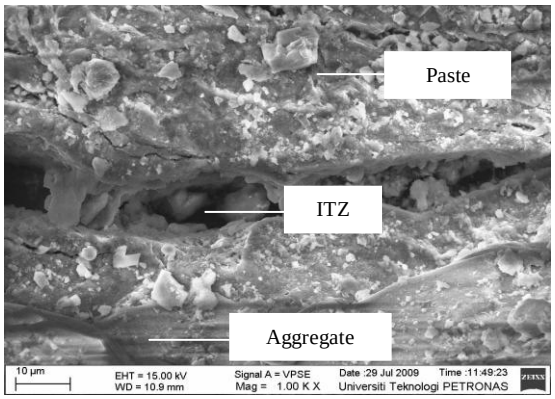

Figure 4. FESEM image of hot gunny curing sample

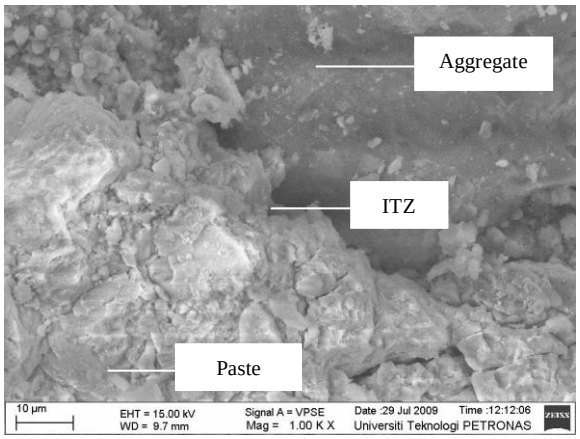

Figure 5. FESEM image of ambient curing sample

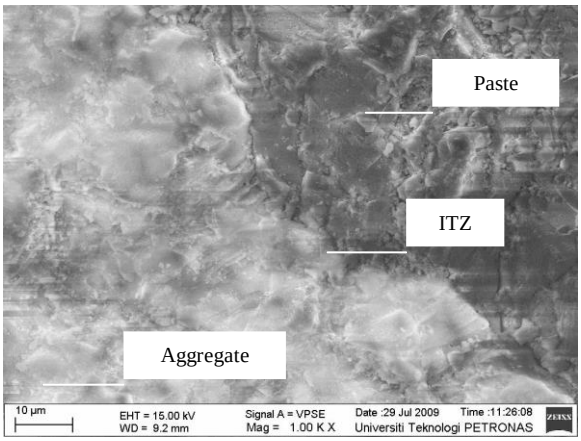

Figure 6. FESEM image of external exposure curing

TABLE 1.
FLY ASH CHEMICAL COMPOSITION

Oxide	Percentages (%)
SiO_2	51.19 %
Al_2O_3	24.00 %
Fe_2O_3	6.60 %
CaO	5.57 %
MgO	2.40 %
SO_3	0.88 %
K_2O	1.14 %
Na_2O	2.12 %

TABLE 2.
MIRHA CHEMICAL COMPOSITION

Oxide	Percentage (%)
SiO_2	88.90 %
MgO	0.72 %
SO_3	0.32 %
CaO	0.63 %
K_2O	3.65 %
Al_2O_3	0.16 %
Fe_2O_3	0.45 %

TABLE 3.
DETAIL OF MIX PROPORTION

Mix Code*	Fly Ash (kg/m^3)	MIRHA (kg/m^3)	Coarse Aggregate (kg/m^3)	Fine Aggregate (kg/m^3)	NaOH (kg/m^3)	Na_2SiO_3 (kg/m^3)	Water (kg/m^3)	Sucrose (kg/m^3)
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

* A: hot gunny curing; B: ambient curing; C: external exposure curing

TABLE 4.
COMPRESSIVE STRENGTH DEVELOPMENT OF GEOPOLYMER CONCRETE

Type of Curing	Code	% Replacement by MIRHA	Compressive Strength (MPa)			
			3 days	7 days	28 days	56 days
Hot gunny Curing	A1	0	4.67	10.53	15.74	16.62
	A2	3	3.43	8.41	12.83	14.96
	A3	5	7.11	11.31	19.01	22.66
	A4	7	3.19	7.01	11.5	12.77
Ambient Curing	B1	0	7.46	14.11	19.73	21.92
	B2	3	6.3	11.05	17.92	19.19
	B3	5	9.38	14.74	25.3	27.28
	B4	7	8.55	11.35	16.75	17.03
External Exposure Curing	C1	0	32.78	44.76	48.88	50.96
	C2	3	19.93	24.96	35.2	36.35
	C3	5	14.14	24.29	28.7	33.62
	C4	7	16.81	30.22	41.34	44.84