Mechanical Properties of MIRHA-Fly Ash Geopolymer Concrete

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Abstract. This paper provides a report about the results of an investigation carried out to understand the effect of Microwave Incinerated Rice Husk Ash (MIRHA) on the mechanical properties of fly ash geopolymer concrete to access the concrete performance development. Fly ash (350kg/m³) and MIRHA (0%, 3%, and 7%) were used as the source materials to replace cement, NaOH and Na₂SiO₃ solutions used as the alkaline liquids for the medium of polymeric reaction. In addition, sugar was used as retarder, as well as three different types of curing regime (ambient, external exposure or oven curing regime). The concrete mixing procedure was adjusted to obtain the proper homogeneity of dry materials and wet ones. In this project, a number of mechanical tests have been conducted including the pull-out test, compressive strength test, flexural strength test, and modulus of elasticity test. It was then observed that the performance of mechanical properties of MIRHA-fly ash geopolymer concrete improved with the use of oven curing as the curing regime for the concrete samples.

Introduction

Concrete is the backbone. It is one of the most widely used construction materials such as in the Hoover Dam, Petronas Towers, and Dames Point Bridge. It is a composite of aggregate, Ordinary Portland Cement (OPC) and water. OPC is the key constituent of concrete, a hydraulic material containing at least two-thirds by mass of calcium silicates (3CaO.SiO and 2CaO.SiO₂).

Since the last three decades, the use of OPC as a binder in a mixture concrete is often criticized by circles concerned with environmental conservation particularly associated with global warming. Here, greenhouse gas glass, such as CO_2 (carbon dioxide), taking place due to human activities, such as the production process cement contributes to the global warming. Of 1 ton of OPC produced, it can produce ± 1 ton of CO_2 [1], which in turn will be released into the air.

This then indicates that the OPC industry is faced by economic, energy and environmental problems (over 6 % of total world-wide CO_2 emissions [2]). Also, it has been reported that many concrete structures, especially those built in corrosive environments, start to deteriorate after 20 to 30 years, even though they have been calculated for more than 50 years of service life [3].

Joseph Davidovits, the inventor and developer of geopolymerization, coined the term "geopolymer" in 1978 to classify the newly discovered geosynthesis producing the inorganic polymeric materials. Environmentally motivated geopolymer applications are based on geopolymer cements with very low CO_2 emission [4, 5].

The geopolymer concrete material is mainly formed by two constituents the source materials and the alkaline liquids. An alumina-silicate material as the main materials for geopolymers should be dominated in silicon (Si) and aluminum (Al). These could be obtained natural minerals such as kaolinite, or clays [6]. Otherwise, by-product materials such as fly ash, silica fume, slag, rice-husk ash, or red mud could be used as the source materials. The option of the source materials for creating geopolymers depends upon some factors such as availability, cost, type of application, and specific demand of the end users. Meanwhile, the alkaline liquids as alkaline activator [7, 8] materials such as NaOH, KOH, Na/K silicates are from soluble alkali metals that generally are Sodium or Potassium based. An alkaline liquid could be reacted with both the silicon (Si) and the aluminum (Al) in the source material and the chemical reaction that takes place in this case a polymerization process to form the geopolymer paste and to bind the aggregates and other un-reacted materials.

On the other hand, more than 100 countries in the world cultivate rice with an annual production of 650.2 million tons [9]. During the milling process of paddy rice, more than 130 million tons of rice husk are obtained as by-product. On combustion, the amount of husk will yield about 26 million tons of ash. In Malaysia, a huge amount of rice husk produced in one year can approximately reach 2.231 million tons. When burnt, about 20% of the rice husk will turn into RHA [10] indicating that 0.1 million tons of RHA can potentially be annually produced in Malaysia.

In Malaysia, rice husk (RH) is an abundant waste material. Burnt (open and/or controlled) RH needs costly disposal in view of its capability to create environmental issues from air pollution to the environment. A proper burning method, in response, is deemed essential purposely to obtain RHA with high reactive silica content. A modern incinerator is then designed to avoid an environmental problem as caused by open burning. One of the modern incinerators, microwave incenerator is proposed to produce amorphous Microwave Incinerated Rice Husk Ash (MIRHA) [11, 12] with high pozzolanic reactivity with one consideration that it can significantly enhance concrete properties. This study is a fundamental study of the use of MIRHA on fly ash geopolymer concrete. It establishes the optimum replacement percentage of fly ash by MIRHA that improves the Geopolymer quality. The knowledge of the use of fly ash together with MIRHA to produce geopolymer, therefore, would be beneficial for both the understanding and the future applications of this material in the construction industries.

Experimental Method

Material

Alkaline activators in this research were obtained from a supplier in Malaysia with a number of specific requirements. NaOH, meanwhile, was supplied by QuickLab Sdn Bhd, Malaysia in pellet form with 99% purity. For all mix proportion, 8 molar NaOH solution was used. Further, obtained from Malay-Sino Chemical Industries Sdn Bhd, Malaysia NaSiO₂ is with proportion of Na₂O: 14.73%, SiO₂: 29.75%, and water: 55.52%.

In the present experimental work, low-calcium (Class F) [13] dry fly ash obtained from Manjung Power Plant, Malaysia was used as the base material. Rice husk obtained from Bernas Milling Plant, Malaysia in this case, was used to produce MIRHA. Firstly, it was dried under direct sunlight to remove its moisture content on purpose to reduce excess smoke from its combustion. Once accomplished, the rice husk was then incinerated in a microwave incinerator at 400°C. The UTP Microwave Incinerator (UTPMI) is the Air Cooled Magnetron system with an overall dimension of $2.3(H) \times 4.0(W) \times 4.0(L)$ and a chamber capacity of 1 m³. MIRHA was ground 2000 times for the fineness in a ball mill. Tabel 1 presents the chemical composition of the fly ash and MIRHA, as determined by x-ray fluorescence (XRF) analysis.

The coarse aggregates sized 20 mm were prepared under saturated surface dry (SSD) conditions. Since commercial water reducing admixture was not suitable for the mixtures in this research, glucose solution was then added into the mixture to delay the setting time during the mixing and casting process.

Ovida composition	Weig	Weight (%)		
Oxide composition	MIRHA	Fly Ash		
Silicon dioxide or silica (SiO ₂)	90.75	51.19		
Aluminum oxide or alumina (Al ₂ O ₃)	0.75	24.0		
Iron oxide (Fe_2O_3)	0.28	6.6		
Calcium oxide or lime (CaO)	0.87	5.57		
Magnesium oxide or magnesia (MgO)	0.63	2.40		
Sodium oxide (Na ₂ O)	0.02	2.12		
Potassium oxide (K ₂ O)	3.77	1.14		
Equivalent alkalis (Na ₂ O + 0.658 K ₂ O)	2.50	1.59		
Titanium oxide (TiO ₂)	0.02	-		
Phosphorous oxide (P ₂ O ₅)	2.5	-		
Manganese oxide (MnO)	0.08	-		

Table 1. Composition of fly ash and MIRHA as determined by XRF (mass %)

Experimental Set up

The mixture proportion was calculated with different amounts of blended source materials to investigate the resultant geopolymer concrete properties. In this case, the constant amounts of NaOH and NaSiO₂ were used throughout the mix proportions. Table 2 describes the details of each mixture. Alkaline solutions were prepared fresh 1 hour before the mixing process to avoid the formation of precipitates in the NaOH solution. The concrete mixing procedure for this project was adjusted to obtain the proper homogeneity of dry materials and wet materials. The procedures were as followos: (i) the coarse aggregates, fine aggregates, MIRHA, and fly ashes were mixed in dry condition for 2.5 minutes. (ii) The alkaline solution of NaOH and Na₂SiO₃, extra water and glucose were added to the mixture. (iii) The mixture was mixed for another 1.5 minutes in wet condition. (iv) The fresh concrete mixture was then cast in 100 mm cubes steel moulds in three layers and compacted using vibrator machine. Three cubes were prepared for each test variable.

Table 2: Mixture Proportion									
Type of Curing Regime	Code	Fly Ash (kg/m ³)	MIRHA (kg/m ³)	Coarse Agg. (kg/m ³)	Fine Agg. (kg/m ³)	NaOH (kg/m ³)	Na2SiO3 (kg/m ³)	Extra water (kg/m ³)	Sugar (kg/m ³)
Ambient Curing	A1	350	0	1200	645	41	103	35	10.5
	A2	339.5	17.5	1200	645	41	103	35	10.5
	A3	315	35	1200	645	41	103	35	10.5
External Exposure Curing	B1	350	0	1200	645	41	103	35	10.5
	B2	339.5	17.5	1200	645	41	103	35	10.5
	В3	315	35	1200	645	41	103	35	10.5
Oven Curing	C1	350	0	1200	645	41	103	35	10.5
	C2	339.5	17.5	1200	645	41	103	35	10.5
	C3	315	35	1200	645	41	103	35	10.5

After mixing, the concrete was left for 24 hours before removing the mould. The hardened samples were then cured in 3 curing conditions: ambient, external exposure, and oven, until the testing days (3, 7, or 28 days). For ambient curing the temperature was between 27°C to 32°C, for external exposure curing, sample was placed in chamber under direct sunlight with temperature 33°C to 40°C and for oven curing, the concrete sample was left in ambient temperature for 1 hour after casting (delay time). The sample subsequently was placed in the oven at 65°C for 24 hours before placed in ambient temperature after the removal of the mould.

Testing

Hardened geopolymer concrete specimens were tested in four destructive tests. Table 3 below shows the detail of the experimental.

Test Type	Standard	Equipment	Age	Sample Size	No. of Samples per Age	Unit
Compression	BS1881-111:1983	Compressive	3,7,28days	100 mm cube	3/mix	N/mm ²
Strength		Machine				
Flexural	BS 1881- 118: 1983	Flexural	28 days	100×100×500mm	3/mix	N/mm ²
Strength		Machine				
Modulus	BS1881-121:1983	Compressive	28days	100mm Φ x	3/mix	N/mm ²
Elasticity		Machine	2	200mm cylinder		

Table 3: Hardened geopolymer concrete test Mixture Proportion

As shown, there is not any particular standard for , there the pull out test. However in a number of literatures, some researchers have used cylindrical concrete sample with an embedded steel bar is to be pulled-out during test. In this study 150 diameters and 300 mm high concrete cylindrical samples with 10 mm diameters of steel bars embedded in the middle were used. It used two approaches for the collation of the embedded length, L. In the first approach the contact area of all diameters of steel bars with concrete was kept constant, whereas in the second approach, the embedment length was 15 kept times the diameter of the steel bar. Fig 1 shows a pull-out test set up. UTM here was applied at the rate of 0.2 kN/s to steel bar until failure happened. Noted here, this test was performed at the age of 3, 7 and 28 days.



Fig.1 Pull out test setup

Result and Discussion

Pull Out

Fig 2 shows the result in the graph of bonding capacity performance for each curing regime. As shown, the addition of MIRHA into the concrete mix proportions in fact is able bring some various effects on the bonding capacity of concrete performance. The interlocking materials here are affected by the mixture composition of the geopolymer concrete, consisting of coarse aggregates, fine aggregates, fly ash particles, and MIRHA particles. The polymeric reaction also affects the bonding capacity of the concrete.



Fig. 2 Bonding capacity of fly ash - MIRHA geopolymer concrete in 3 curing conditions

The bonding capacity performance increases throughout the age of each sample. From the results, oven curing regime samples with different percentage of MIRHA have the highest bonding capacity performance for the samples at the early age and for 28 days. In contrast, the samples from ambient curing regime for all MIRHA content have the lowest development of bonding capacity performance for the samples at early age and for 28 days.

The curing regime surrounding temperature plays an essential role in the polymeric reaction process. The polymeric reaction of geopolymer concrete is increasing with the increasing of the surrounding heat. Thus the development of the geopolymer concrete bonding capacity performance will increase as well. For oven curing regime, the obvious slight decrease of bonding capacity performance during the early age of concrete shows the instability of the polymeric reaction process. This is due to the sample removed from oven with high temperature to ambient condition with low temperature, leading to the loss of heat and affecting the polymeric reaction of the geopolymer concrete.

Compressive Strength

The results obtained from compressive strength tests show the polymeric reactions in geopolymer concrete with various curing treatments. Fig. 3 describes the compressive strength development of geopolymer concrete for all curing regimes.



Fig 3: Compressive strength of MIRHA-fly ash geopolymer concrete in 3 curing conditions.

The compressive strength increases throughout the age of each sample. From the results, the oven curing regime samples with the different percentages of MIRHA have the highest compressive strength development for the samples with early age and those for 28 days. A blended source material with 97% fly ash and 3% MIRHA, meanwhile, provided some better results compare to other samples in the oven curing as shown in Fig 3. The compressive strength for a blended sample increased by 16 to 26% compared to a non-blended sample of the same curing regime. It shows that the different amounts of Al-Si materials in fly ash and MIRHA affected the properties of geopolymer concrete.

Samples from ambient curing regime for all MIRHA contents, meanwhile, have the lowest development of compressive strength performance for the samples with early age and for those 28 days. For ambient and external exposure curing regime, the addition of MIRHA to the concrete mix is not favorable to the strength development of concrete when compared to control sample. MIRHA contains high SiO_2 content and the presence of SiO_2 content in high amount interfere with the polymeric process, which will alter the microstructure of concrete and affect the concrete. The unstable polymeric reaction will affect the compressive strength development of the concrete. The compressive strength has a definite relationship with all of other concrete properties. In addition to this, the improvement of compressive strength will improve the other concrete properties.

It was due to MIRHA particles have the large amount of amorphous and the high SiO_2 which affected the significantly higher compressive strength and induced the faster polymerization reaction.

Modulus of Elasticity

The modulus of elasticity is a mathematical description of the tendency of concrete to deform elastically. In this study, it is the initial tangent modulus or Young's modulus of the concrete beam sample. Fig. 4 shows the modulus elasticity of MIRHA-fly ash geopolymer concrete for 28 days.



Fig 4: Modulus of Elasticity of fly ash - MIRHA geopolymer concrete for 28 days

As shown in Fig. 4, the lowest modulus elasticity takes place in ambient curing sample for all MIRHA contents. Here, with the addition of MIRHA content, the modulus elasticity of ambient curing regime increases. For the external exposure, the addition of MIRHA content gives various effects on the modulus of elasticity and for oven curing regime is averagely higher than other curing regime but without any significant increase to the modulus of elasticity with addition of different percentages of MIRHA.

Sample A1 has the lowest value of modulus elasticity at 906 N/mm² and sample A2 has the highest value at 3792 N/mm². The modulus elasticity increases with the increase of compressive strength but minus precise relationship form. The increase in modulus elasticity is progressively lower than that of compressive strength. That become the reason why the modulus of elasticity is tested for 28 days.

Neville (1995) stated that modulus elasticity was found not to be affected by curing [14]. This is true for normal concrete using Portland cement but for geopolymer concrete, the curing regime will affect the modulus of elasticity as shown from the result. Polymeric reaction progress is affected by the curing regime types as polymeric reaction is affected by the temperature of the curing regime. High curing regime temperature will accelerate the polymeric reaction of geopolymer concrete.

Flexural Strength

Flexural strength test was conducted to determine the ability of the MIRHA-fly ash geopolymer concrete beam to resist failure under loads that cause it to bend. This test was conducted for 28 days in light of the low development of flexural strength. Fig. 5 shows the column chart of the flexural strength data for 28 days for ambient, external exposure, and oven curing regime.



Fig 5: Flexural Strength of fly ash - MIRHA geopolymer concrete for 28 days

Generally, MIRHA-fly ash geopolymer concrete samples have a higher flexural strength compare to the normal fly one. It is due to the fact that this may mainly owe to OH- ion of NaOH used in the reaction with Si component in fine MIRHA purposely to link the new monomer of Si-O-H better than normal fly ash geopolymer concrete. MIRHA particles were abundant and affected to the free OH- ion of NaOH still left in the matrix of fresh geopolymer mortar.

The flexural strength also affected by the curing regime types as the surrounding heat assists the strength development. MIRHA content also affects the flexural strength as high amount of SiO_2 in MIRHA interfere with polymeric reaction of geopolymer concrete.

Conclusion

The addition of different percentages of MIRHA into the concrete mixture proportions in fact is able to provide various effects the development of bonding capacity, compressive strength, modulus of elasticity, and flexural strength performance of fly ash - MIRHA geopolymer concrete. Overall, samples from oven curing regime with the addition of different percentages of MIRHA have the highest mechanical properties performance for all concrete ages. By contrast, samples from ambient curing regime with the addition of different percentages of MIRHA have the lowest mechanical properties performance for all concrete ages. The performance of bonding capacity, compressive strength, modulus of elasticity, and flexural strength of MIRHA-fly ash geopolymer concrete is improved using oven curing as the curing regime for the concrete samples.

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