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Study on Dry-Wet Cyclic Resistance of Geopolymer Mortars Using Blended Ash from Agro-Industrial Waste

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ABSTRACT

This paper presents the dry-wet cyclic resistance of geopolymer mortars prepared from the combination of palm oil fuel ash (POFA) and pulverized fuel ash (PFA) from agro-industrial waste, as cement replacement and activated by alkaline solution. Alkaline solution was prepared by combining sodium silicate and sodium hydroxide. The optimum mix proportions of geopolymer mortars with PFA: POFA mass ratio of 70:30 was used together with alkaline solution. The ratio of sodium silicate solution-to-sodium hydroxide solution by mass was 2.5:1. The mass ratio of sand to blended ashes was 3:1. Tests were carried out using 70x70x70 mm cube geopolymer mortar specimens. Ordinary Portland cement (OPC) mortar was also prepared as control specimens and cured in water for 28 days. Geopolymer mortar specimens were cured at room temperature (28ºC) for 28 days and heat cured at 90ºC for 24h, respectively. Dry-wet cycle test was conducted to see the resistance of geopolymer mortars towards aggressive weather conditions. The test results revealed that geopolymer mortars showed high resistance to aggressive weather changes as compared to ordinary OPC mortar due to the elimination of cement in the mixture.

Keyword. geopolymer, palm oil fuel ash, pulverize fuel ash, alkaline solution, dry-wet cyclic

INTRODUCTION

The ordinary Portland cement (OPC) still continues to be the most commonly used in construction field. Many studies have shown that OPC gives poor performance in resistance to extreme climate and chemical condition. Moreover, it processes and releases a large amount of green house gas, i.e. carbon dioxide (CO_2) into the atmosphere. Most of the research carried out involves the development of geopolymers as a potential large-scale replacement for concrete produced from Portland cement (Allouche, 2012). This is due to geopolymers' lower carbon dioxide production emissions, greater chemical and thermal resistance and better mechanical properties at both ambient and extreme conditions.

Geopolymers are generally formed by reaction of an aluminosilicate powder with an alkaline silicate solution at roughly ambient conditions. Metakaolin is a commonly used starting material for laboratory synthesis of geopolymers, and is generated by thermal activation of kaolinite clay. Geopolymers can also be made from sources of pozzolanic materials, such as fly ash from coal. Most studies on geopolymers have been carried out using natural or industrial waste sources of metakaolin, fly ash and other aluminosilicates

Geopolymer is a new material which is being used for construction all over the world. (Davidovits, 1990; 1994, a; b; c; 1999) proposed that an alkaline liquid could be used to react with the silicon (Si) and the aluminum (Al) in a source material of geological origin or in by-product materials such as fly ash and rice husk ash to produce binders. Because the chemical reaction that takes place in this case is a polymerization process, he coined the term 'Geopolymer' to represent these binders.

Most of the researches in geopolymer technology only used pulverized fly ash (PFA) as a source material to replace cement (Hardijitro, 2005; Rangan, 2008 (a); 2008 (b); 2009). Palm oil shell and husk, when burnt, is found to contain a high percentage of silica which is one of the main constituents in producing geopolymer. It is an agricultural waste and one of the pozzolan materials that has been successfully used in the improvement of strength and durability of concrete (Awal, 1997a; 1997b; 1996). Whereas increased use of palm oil fuel ash (POFA) is evident, much of the POFA is disposed in land filling, still huge volume of unutilized POFA. It was considered to utilize POFA blending with PFA as a source material in the preparation of geopolymer mortars.

As a new material for construction, very little research has been conducted on the durability of geopolymer mortar or concrete particularly with reference to weather resistance (Bhutta, 2001). The durability of concrete is an important requirement for the performance in aggressive environments throughout its design life period. In the present study, the dry-wet cyclic resistance of geopolymer mortars was examined keeping in view the equatorial Malaysian climate where intense downpour is followed by scorching hot sunshine.

MATERIALS

Blended Ash and Fine Aggregate. Lignite PFA from Kapar power station, Selangor, Malaysia was used. POFA was obtained from burning of palm oil shell and husk (in equal volume) at 940℃ from a Kahang mill, Kluang, Johor. The PFA and POFA have a mean particle size of 45μm with percentages retained 92.9% and 90% on the sieve. The obtained ashes were greyish and the losses on ignitions (LOI) were 0.11% for PFA and 20.9% for POFA. The chemical compositions of PFA and POFA are given in Table 1. Local crushed granite sand with a specific gravity of 2.62 was used for making mortar.

Table 1. Chemical compositions (%) of PFA and POFA

Material	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	P_2O_3
PFA	46.7	35.9	5.0	3.9	0.8	0.6	0.5	0.4
POFA	53.5	1.9	1.1	8.3	4.1	1.3	6.5	2.4

Alkaline Solutions. To activate the blended ash, commercial grade sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) solutions were used as alkaline activator. Distilled water was used to dissolve sodium hydroxide pellets to prevent any effect of unknown contaminants. The mass of sodium hydroxide solids in a solution varies depending on the concentration of the solution. The range of sodium hydroxide concentration used in this study was 14 molar. In order to improve the workability, a super plasticizer was added to the mixture.

TESTING PROCEDURES

Preparation of Specimens. The optimum mix proportions was used to prepare geopolymer mortars as shown in Table 2. All geopolymer mortar specimens were prepared with sand to blended ash ratio of 3:1, whereby the sand was prepared to saturated surface dry condition. The concentration of alkaline solution used was 14 molar. The ratio (by mass) of sodium silicate (Na_2SiO_3) to sodium hydroxide (NaOH) was 2.5:1. The optimum PFA to POFA ratio was 70:30 in the mix proportions. Blended ash, super plasticizer (powder form, SP) and the aggregates were first mixed together dry in a mixer for three minutes. The alkaline solution was then added to dry mixture and mixing continued for another four minutes. The mixing was carried out at room temperature of approximately 28℃. The flow of geopolymer mortars was determined and fixed in the range of 130±5 according to ASTM C 1437. The fresh mortar was cast into 70x70x70 mm cubic molds and compacted by the usual methods used in the case of Portland cement mortar or concrete. The specimens were wrapped with plastic sheets to prevent from moisture loss.

For OPC mortar as control specimens, the ratio of cement to sand was 1:3 with water-cement ratio of 0.45%. The mortar was mixed and the flow was determined in accordance with the procedures given in ASTM C 1437 and fixed to 130±5.

Table 2. Optimum mix proportions of geopolymer mortar

Mix proportions, kg/m^3				**(%)**	
Blended Ash	Alkaline Solution	Sand	SP	Liquid / Blended Ash	Water/ Liquid
527	237	1586	5.27	0.45	0.27

Curing. The test specimens of geopolymer mortars were (i) heat-cured at 90℃ for 24-h plus 7-d dry cure, and (ii) placed at room temperature approximately 28℃ for 28-d. OPC mortar specimens were placed into water for curing for 28-d.

Dry-wet cyclic resistance test. The deterioration of concrete due to the dry-wet cycles under various weather conditions may be occurred because of the difference in the dry shrinkage and coefficient of thermal expansion of the cement mortar as a binder and aggregates. Dry-wet cyclic resistance test was considered to see the performance of geopolymer mortars under harsh tropical climate condition (Kajio, 2004). The test was employed keeping in view the equatorial Malaysia climate where intense downpour is followed by scorching hot sunshine. All specimens went through 30 dry-wet cycles. The details of dry-wet cycle test are given in Table 3 and Fig. 1.

Table 3. Test conditions for cyclic wetting and drying

Specimen size	70 x 70 x 70 mm
Wet condition	In water (20°C)
Dry condition	In oven (40°C)
Cycle	3 days in dry condition –1 day in wet condition –2 days in dry condition –1 day in wet condition (This means 2 cycles)
Test end	Total 30 cycles

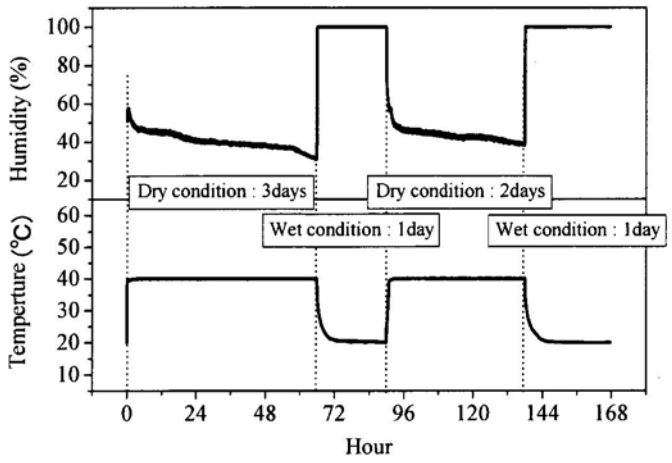

Figure 1. Cycles of wetting and drying for automatic test machine

Ultrasonic Pulse Velocity (UPV) Test. After every two dry-wet cycles, specimens went for ultrasonic pulse velocity to determine any deterioration such as external and internal cracks**.**

UPV apparatus normally measures the travel time of longitudinal waves to determine the pulse velocity. The test apparatus consists of an ultrasonic pulse generator that is combined with a transmitter. The apparatus is coupled to the test specimen by two independent transducers that transmit and receive the generated ultrasonic pulse. A processor measures the time for this pulse to travel between the two transducers and calculated the velocity of the pulse through the material being tested. The density of the material, void, cracks and other imperfections affect the velocity of the pulse. Variations in the measured velocity thus indicate the presence of voids, cracks or imperfections in the path measured. When an ultrasonic pulse travelling through mortar or concrete intersects a crack or void in the concrete mass, its measured velocity will be reduced because of travelling the air in this void or crack. The amount of reduction will depend on the width of the crack as well as whether the crack is filled with water or debris. In addition, the pulse will also be deflected or bypass the crack or void at the same velocity as the original velocity. This is shown in Fig. 4. If the crack is very narrow with sides virtually touching or filled with water and/or debris, the pulse will travel through it with only a slight reduction in velocity and the crack will be difficult to measure. The instrument thus to be extremely sensitive to detect micro-cracks.
Pulse velocity was calculated based on following equation:

$$\text{Pulse velocity (m/s)} = \frac{\text{Specimen size (m)}}{\text{UPV time travel(s)}} \quad (1)$$

Figure 2 illustrates different conditions that may be encountered when testing an element. At the top (a), the path between the transducers is through solid concrete, and the travel time would be the shortest. Below (b) that is the case where there is an internal pocket of porous concrete, such as honeycombed concrete. The pulse is scattered as it travels though the contiguous portions of the honeycombed concrete. As a result, the travel path is longer and the pulse travel time is longer. In the next case (c), the transducers are located so that the direct travel path is near the edge of a crack. The pulse cannot travel across a concrete-air interface, but it is able to travel from the transmitter to the receiver by diffraction at the crack edge. Because the travel path is longer than the distance between the transducers, the apparent pulse velocity is lower than through sound concrete. In the lowermost case (d), the pulse is reflected completely by the crack, and travel time is not measurable

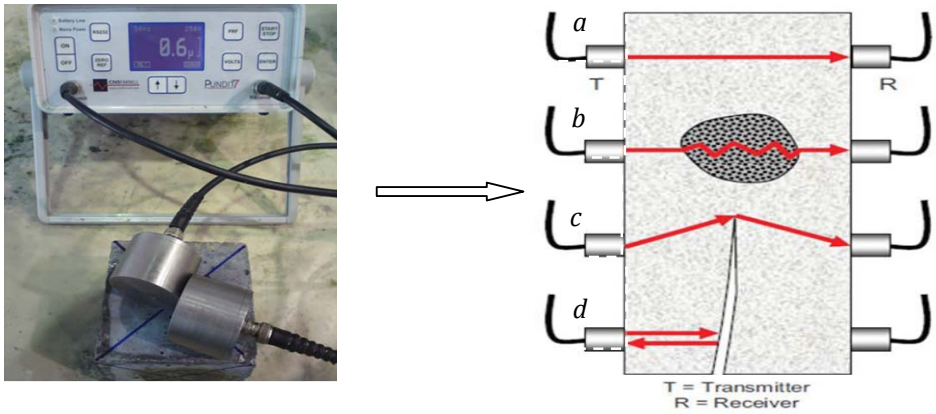

Figure 2. Different conditions that may be encountered when testing an element

Evaluation. The dry-wet cyclic resistance was evaluated based on visual observation, the mass change, ultrasonic pulse velocity and the residual compressive strength of the test specimens after exposed dry-wet cycles. At the end of every two dry-wet cycle, a total of three specimens were tested for ultrasonic pulse velocity. The residual compressive strength test was conducted at 0, 15 and 30 cycle and was calculated based on the following equation:

Residual compressive strength (%) = [B/A] x 100 (2)

where A = Initial compressive strength before exposure
B = Compressive strength after dry-wet exposure

RESULTS AND DISCUSSION

Visual Observation and Mass change. With visual observation, geopolymer mortars remain structurally intact without any visible cracks or deterioration at the surface of geopolymer mortars, however, hair cracks are found at the surface of OPC mortar at the end of dry-wet cycle test as shown in Fig. 3.

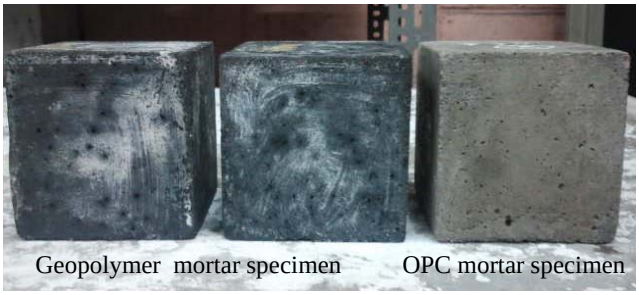

Figure 3. Appearance of specimens after dry-wet cyclic test

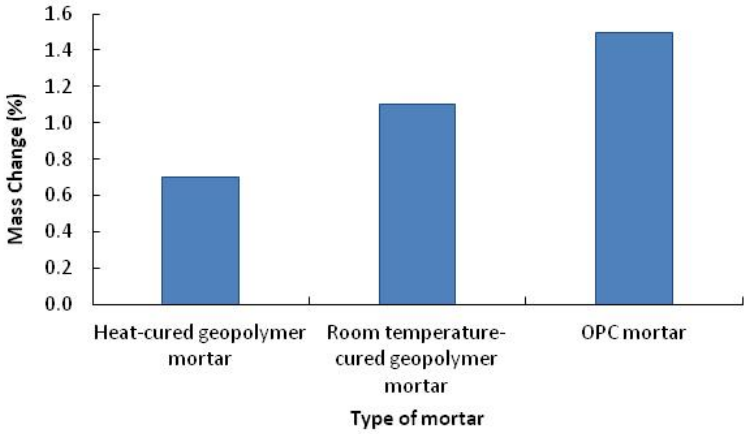

Figure 4. Mass change of mortar specimen exposed to dry-wet cycle

The mass of specimens was slightly increased from initial mass. The mass change of specimens exposed to dry-wet cycles is given in Figure 4. The increments in mass for heat-cured geopolymer mortar and room temperature-cured geopolymer mortar were 0.7%, 1.1%. The OPC mortar showed mass change of 1.5% which was higher than geopolymer mortars. The increment in mass was due to the water absorption property of each mortars.

Ultrasonic Pulse Velocity (UPV). Figure 5 represents UPV vs. dry-wet cycle of mortars. It is seen that there is marked difference between the pulse velocities of hear-cured, room temperature-cured geopolymer mortars and OPC mortar. As expected, OPC mortar specimens have a lower pulse velocity than the higher pulse velocity values for geopolymer mortar specimens, particularly for heat-cured geopolymer mortar specimens. This could be attributed to the fact that under heat curing there is a higher possibility of fast and complete geopolymerization in geopolymer mortar which leads to a denser mortar, and thus to a higher pulse velocity. Evidently there was no internal damage such as microcracks appeared inside the geopolymer mortar specimens during 30 dry-wet cycles. As seen in Figure, a noticeable variation in pulse velocities was observed when the number of dry-wet cycles increases. The huge variation in pulse velocity was seen in Figure 4 during dry-wet cycles that could have occurred because of the difference in the dry shrinkage and coefficient of thermal expansion of the heat-cured, room temperature-cured geopolymer mortars and OPC mortars. Lower pulse velocity values in OPC mortar specimens which indicated that the microcracks might occur in the specimens during 30 dry-wet cycles and could lead to reduction in compressive strength of the specimens.

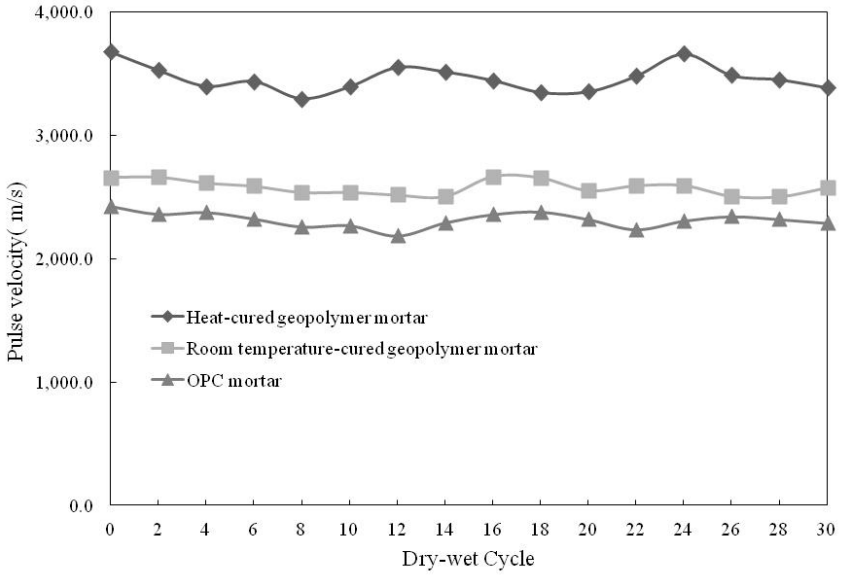

Figure 5. Ultrasonic pulse velocity vs. dry-wet cycle of mortars

Residual Compressive Strength. To evaluate the effect of dry-wet cycles, the compressive strength test was conducted at the following cycle intervals of 0-cycle**,** 15-cycle and 30-cycle. Figure 6 shows the effect of dry-wet cycles on the residual compressive strength of geopolymer and OPC mortars.

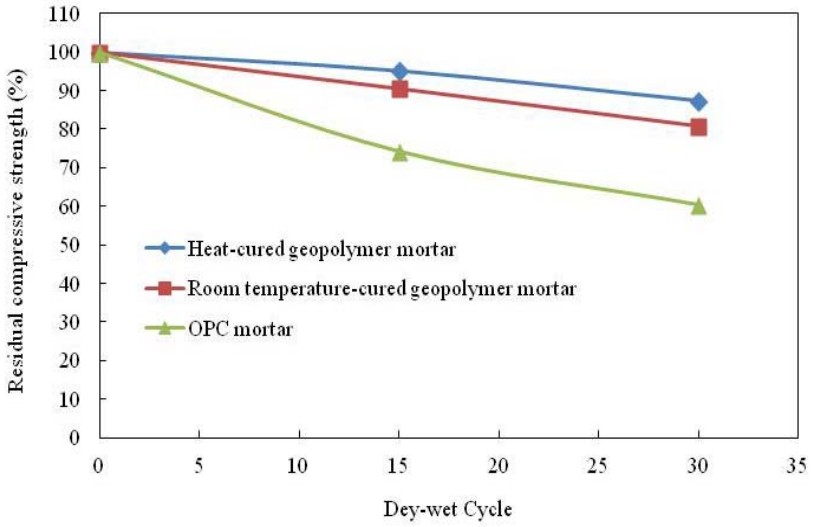

Figure 6. Residual compressive strength vs. dry-wet cycle

Regardless of type of mortar, the residual compressive strength is gradually decreased with increase in dry-wet cycles. The rate of reduction in the residual compressive strength in geopolymer mortars was much lower than OPC mortars. OPC mortars showed more than 40% reduction in the residual compressive strength after 30 dry-wet cycles. This rate of reduction in residual compressive strength was significant higher than geopolymer mortars. This is attributed to micro cracks due to differences in the dry shrinkage and coefficient of thermal expansion of OPC mortar. The residual compressive strength of heat-cured geopolymer was about 8% which was lower than room temperature-cured geopolymer mortar of more than 15%. Heat-cured geopolymer mortar exhibited less residual compressive strength due to complete geopolymerization process which produce more dense and strong bonding structure. As mentioned above, the higher UPV values were obtained from geoopolymer mortars during 30 dry-wet cycles, which indicated the effect of the filling and packing capacity of blended ash (PFA+POFA) on UPV values. Finer particles of blended ash filled the micro pores in the geopolymer paste and improved the strength and durability.

CONCLUSIONS

1. Geopolymer mortars remain structurally intact without any visible cracks or deterioration at the surface of geopolymer mortars, however, hair cracks are found at the surface of OPC mortar at the end of dry-wet cycle test. The increments in mass for heat-cured geopolymer mortar and room temperature-cured geopolymer mortar were 0.7%, 1.1%. In contrast, OPC mortar showed mass change of 1.5% which was higher than geopolymer mortars.
2. OPC mortar specimens have a lower pulse velocity than geopolymer mortar specimens, particularly for heat-cured geopolymer mortar specimens. This could be attributed to the fact that under heat curing there is a higher possibility of fast and complete geopolymerization in geopolymer mortar which leads to a denser mortar, and thus to a higher pulse velocity.

3. OPC mortars showed more than 40% reduction in the residual compressive strength after 30 dry-wet cycles. This rate of reduction in residual compressive strength was significantly higher than geopolymer mortars. The residual compressive strength of heat-cured geopolymer was about 8% which was lower than room temperature-cured geopolymer mortars of more than 15%.
4. As a result, geopolymer mortars showed good weather resistance (dry-wet cycles) due to geopolymer binder, particularly, heat-cured geopolymer exhibited good performance.

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