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Chemical Attack of Malaysian Pozzolans Concrete

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Abstract

Malaysia produces more than 10 million tonne of by-product from industrial sector per year. As the environmental impact concerns, more than half of the by-product can be recycled to be used as construction materials. One of them is pozzolan, a cement replacing material that can be used to enhance the properties of the concrete. This paper studies the chemical attack to local prozzolans concrete. The parameters studied include weight loss, length change, and residual strength of local pozzolans concrete after been exposed to severe environment. The specimen were tested under normal room temperature, exposed to magnesium sulfate, MgSO4 ● 2H2O where both sulfate attack and acid attack take place. Two series of pozzolans which is Pulverized fly ash (PFA) and Palm oil fuel ash (POFA) were identified. Another series of admixture, Quarry Dust (QD) from quarry waste that contain high amount of silica content also been carried out. Each series will study the effect of cement replacement percentage of 5%, 10% and 15%. The parameters were compared to conventional ordinary Portland cement (OPC) concrete as control mix. Accelerated testing was conducted at 3, 7, 28, 56 and 90 days. The result shows that the local pozzolans concrete were found to be in good resistance against sulfate attack compared to conventional concrete. Compared to all series conducted, series of PFA replacement gave the best resistance followed by POFA and QD replacement series.

Keywords: pozzolans, weight loss, length change, residual strength, pulverized fly ash

1 INTRODUCTION

As the world progresses towards industrialization and modernization, it causes the pollution such as air pollution. One of the approaches that can contribute to green environment is by using recycled wastes. As indicated in RMK9, Malaysia will be leading in bio-technology in the South-East Asia region, researchers should think of managing its waste. Concrete is the most widely used as construction materials can play its role by replace its major constituent to recycle materials, say cement replacing materials or pozzolans in order to replace Ordinary Portland cement (OPC). OPC Type I is the most common cement used in general concrete construction when there is no exposure to sulphates in the soil or in groundwater.

Local Malaysian pozzolans which is combustion by-product such as palm oil fuel ash, rice husk ash and fly ash are of interest to scientist and engineers. Researchers have been investigating the use of pozzolans in construction materials especially as the concrete additive in or cement replacement materials in order to better manage our waste as well as to improve properties of construction materials. Neville, A.M. (1996) reported that by replacing pozzolan up to 10% can produce optimum strength of concrete. By reusing the waste products from industries, the source of the pollutions can be reduced, helps to reduce the cost of waste management and make our environment more sustainable (N.Ali et al, 1996).

Pozzolan as an amorphous or glassy silicate material that reacts with calcium hydroxide formed during the hydration of Portland cement in concrete. The substance that contribute to the strength of the concrete called calcium silicate hydrate (C-S-H). Calcium hydroxide will reduce the strength of the concrete. Pozzolans contains silica that react with calcium hydroxide in concrete to form extra calcium silicate hydrate compound and diminish calcium hydroxide, further strengthening the concrete due to increase of C-S-H compound and making it stronger, denser, and durable during its service life (Neville, A.M., 1981). Pozzolans also serve to reduce the permeability of the concrete, which helps to make it more resistant to deterioration and swelling associated with various exposure conditions.

$$\underset{\text{Dicalcium silicate}}{C_2S} + \underset{\text{water}}{H_2O} \rightarrow \underset{\text{calcium silicate hydrate}}{C-S-H} + \underset{\text{calcium hydroxide}}{Ca(OH)_2}$$

$$\underset{\text{Calcium hydroxide}}{Ca(OH)_2} + \underset{\text{silica oxide}}{SiO_2} \rightarrow \underset{\text{calcium silicate hydrate}}{C-S-H} + \underset{\text{water}}{H_2O}$$

Sulfate attack on concrete represents a special case of chemical attack. Seepage water, ground water and alkali soil which contains a high sulfate salt content (MgSO4, NaSO4 and CaSO4) can lead to disintegration of concrete. The resistance of concrete to sulfate attack can be tested by storing the specimen in solution of sodium or magnesium sulfate or in a mixture of the two. The resistance to sulfate attack can be improved as reported by Aimin, Xu, (1997); by (i) Adding or partial replacement of cement by pozzolan (ii) Using high pressure steam curing.

This paper will study the effect of chemical attack on few series of concrete identified. Admixture including combustion by-product or known pozzolans was added into the concrete as cement replacement to enhance the properties of concrete. Pulverized fly ash (PFA) and Palm oil Fuel ash (POFA) were identified as potential pozzolans as cement replacement to be commercialized due to its abundant amount were determined. Another potential admixture from quarry industry which is quarry dust also included as one of the concrete series to study its behaviour against chemical resistance. Quarry Dust (QD) was selected in this research due to high content of silica and there is huge amount and normally treated as disposal waste and it may have potential to e marketed. However, since this product is not due to the combustion process, the reactive silica might not be as good as pozzolans to produce a denser concrete. Each of the series above consists of 5%, 10% and 15% of cement replacement to study the effect of different percentage against chemical resistance. The results obtained were compared to Ordinary Portland cement (OPC) as control concrete. The parameter used in this study includes weight loss, length change and residual strength. Accelerated test was carried out where the samples were immersed in the sulfate solution about 24 hours after casting to indicate the worst scenario. The entire samples were carried out up to 90 days.

2 MECHANISMS OF SULFATE ATTACK

According to ACI's Guide to Durable Concrete (ACI 1992), there are two mechanisms that can be considered to be sulphate attack which the formation of gypsum and formation of ettringite. Cohen et al. (1990) reported that at low SO42- concentrations (less than 1000 mg SO42- / litre), deterioration due to formation of ettringite while on the other hand, at high concentration, formation of gypsum is the main cause of deterioration. Both of these reaction products are believed to damage concrete by increases in overall solid volume. Based on Skalny, J. et. al. (2002), Chemical sulfate attack is considered to be the result of chemical reactions involving sulfate anion, SO42-. Example of such reaction is formation of ettringite from monosulphate and gypsum, according to the following stoichiometric reaction:

$$\underset{Monosulphate}{C_4ASH_{12}} + \underset{Gypsum}{2CSH_2} + \underset{Water}{16H} \rightarrow \underset{Ettringite}{C_6AS_3H_{32}}$$

where $C = CaO$, $A = Al_2O_3$, $S = SO_3$ and $H = H_2O$. The reaction is known to result in an increase in solid volume of the system, and may lead to the expansion of concrete.

External sulfate attack is caused by a source external to concrete. Such sources include sulfates from ground water, soil, solid industrial waste, and fertilizers, or from atmospheric SO3, or from liquid industrial wastes. Some of the visible examples of damage include spalling, delamination, macrocracking and possibly loss of cohesion. Due to removal from the cement paste of calcium hydroxide causing leaching of calcium and hydroxyl ions out of the system, thus a possible decrease in the alkalinity (pH) of the paste or sulfonation of the Ca2+ ions to form potentially expansive compounds such as ettringite or gypsum. The interaction between hydrated cement paste and a magnesium sulfate solution is a reaction of the sulphate with calcium hydroxide of the paste, yielding magnesium hydroxide [$Mg(OH)_2$] and calcium sulfate in form of gypsum [$CaSO_4 . 2H_2O$]:

$$\underset{Magnesium\ \sup ahate}{Mg^{2+} + SO_4^{2-}} + \underset{paste}{Ca(OH)_2} + \underset{water}{2H_2O} \rightarrow \underset{Magnesium\ hydroxide}{Mg(OH)_2} + \underset{Gypsum}{CaSO_4 \cdot 2H_2O}$$

However, according to Bensted, J. et al. (2002), they state that formation of ettringite or gypsum not necessarily causing expansion. Even if in some cases expansion does occur, it does not necessarily lead to cracking or significant damage to concrete. They also state that it is difficult to develop accurate, reliable, and reproducible test methods and standards for deterioration processes like sulfate attack.

3 PREPARATION OF SAMPLES

3.1 MATERIALS

Pozzolanic materials

There are three local waste products used in this study as admixtures; namely Palm Oil Fuel Ash (POFA), Pulverized Fly Ash (PFA) and Quarry Dust. POFA produced from husk fibre and shell of palm oil burning by generation plant boiler which the burning energy is used to generate electricity for oil extraction process. Both PFA and POFA produced from burning process, therefore this product better known as pozzolan. Another non-pozzolan admixture which contain high amount of silica content, i.e., Quarry Dust produced directly by the rock boulder burst of granite or original aggregate at quarry. Fly ash is the finely divided mineral residue resulting from the combustion of pulverized coal in electric generating plants. All the materials are collected and physical properties were determined before design mixes were carried out. The physical properties of all admixtures were determined and shown in Table 1.

Table 1: Physical Properties of admixtures and OPC

Physical Properties		POFA	Fly Ash	Quarry Dust	OPC
Consistency and setting times	Water content (%)	-	28.1	-	25.6
(10% pozzolans and 90% OPC)	Initial Setting (h:m)	-	3:14	-	2:15
	Final Setting (h:m)	-	4:25	-	4:05
Blaine specific surface (cm^2/g)		5190	3980	-	3140
Specific Gravity		2.22	2.25	2.65	3.15

Other concrete mix components

Crushed gravel and river sand were used as coarse and fine aggregates respectively in this concrete mix. Their physical properties are shown in Table 2. Ordinary Portland cement was used as control concrete series. In order to compensate the requirement of water content (low w/c ratio) for workability and yet producing optimum strength concrete, an admixture of water-reducing agent or superplasticizers were used. In this study, sulfonated, naphthalene-formaldehyde condensate-type superplasticizer was added to the batch during mixing.

Table 2: Physical properties of Aggregates used

Physical properties	River sand	Crushed stone
Specific gravity (SSD condition)	2.79	2.53
Moisture content (%)	1.33	0.42
Water adsorption (%)	1.65	0.64
Bulk density (kg/m^3)	1534	1445
Fineness Modulus	3.32	-

Magnesium Sulphate

Magnesium sulphate of magnesium sulphate heptahydrate is a chemical compound containing magnesium, with the formula MgSO4.7H2O. Magnesium sulphate (MgSO4) without water of crystallization is available diluted to form into a 0.1% solution.

 

Figure 1: Magnesium sulphate crystal (Left) Curing tank contains samples immered in magnesium sulphate solution (Right)

TEST SAMPLES

Four series of samples were carried out in this study as shown in Table 3. Data collection will be done at the ages of 3rd, 7th, 28th, 56th, and 90th days. On the day 1, initial data were obtained and all the specimens were immersed in solution of $MgSO4.7H2O$ until the day of testing. Data will be carried out from the cube testing (100 mm x 100 mm x 100 mm cube samples) which consists of parameter length change, weight loss and residual strength.

Table 3: Series of Samples

Concrete mixes	Compositions	Mixture Numbers
Series 1 (control mix)	100% OPC	OPC
Series 2 (PFA replacement series)	95% OPC + 5% PFA 90% OPC + 10% PFA 85% OPC + 15% PFA	5R PFA 10R PFA 15R PFA
Series 3 (POFA replacement series)	95% OPC + 5% POFA 90% OPC + 10% POFA 85% OPC + 15% POFA	5R POFA 10R POFA 15R POFA
Series 4 (QD replacement series)	95% OPC + 5% QD 90% OPC + 10% QD 85% OPC + 15% QD	5R QD 10R QD 15R QD

The mix proportions were conducted based on Department of Environmental Method. The mix is based on the target strength obtained as 40 N/mm2 with constant water-cement ratio of 0.55. The proportions of concrete mixes are summarized in Table 4.

Table 4: Mix proportion of concrete

	Mix Number	w/(binders)	Quantities, kg/m^3						
			Water	OPC	PFA	POFA	QD	Fine aggregates	Coarse Aggregates
Series 1	OPC	0.55	225	400	-	-	-	770	1020
Series 2	5R PFA	0.55	225	380	20	-	-	770	1020
	10R PFA	0.55	225	360	40	-	-	770	1020
	15R PFA	0.55	225	340	60	-	-	770	1020
Series 3	5R POFA	0.55	225	380	-	20	-	770	1020
	10R POFA	0.55	225	360	-	40	-	770	1020
	15R POFA	0.55	225	340	-	60	-	770	1020
Series 4	5R QD	0.55	225	380	-	-	20	770	1020
	10R QD	0.55	225	360	-	-	40	770	1020
	15R QD	0.55	225	340	-	-	60	770	1020

4 RESULTS

The physical properties of palm oil clinker, river sand, gravel, cement and fly ash were conducted for mix design data purposes. Properties of fresh concrete; i.e., workability were measured throughout all the series mixes. The fresh concrete properties are shown in Table 5.

Table 5: Fresh concrete properties

Series	**Slump (mm)**			
Percentage replacement of pozzolans	**0%**	**5%**	**10%**	**15%**
Series 1- Control mix	43	-	-	-
Series 2 – PFA Replacement Series	-	10	90	15
Series 3 – POFA Replacement Series	-	52	15	50
Series 4 – QD Replacement Series	-	10	10	70

As mentioned above, there are three parameters been assessed; i.e., residual strength, weight change and length change. Residual strength which is the compressive strength after exposed to sulfate attack follows the BS 1881: Part 116: 1983. The parameters of weight change and length change (expressed in volumes) is a relative study with initial data obtained on the day 1 after been exposed to sulfate solution and expressed in percent (%). Three cubes will be tested for each series of concrete mixes at each age of the cube specimens.

All the pozzolans concrete data will be compared with control concrete and determination of pozzolans replacements percentage gave the best optimum resistance against sulfate attack.

The experiments were performed at Material Lab, Universiti Tun Hussein Onn Malaysia.

5 DISCUSSIONS

Magnesium sulfate initially reacts with the hydrated calcium aluminates and calcium sulphoaluminate was formed together with magnesium hydroxide as described in the mechanism expressed above. Calcium sulphoaluminate itself unstable in the presence of magnesium sulfate solution and by the continued action of this salt was finally decomposed again to form gypsum, hydrated alumina and magnesium hydroxide. The external skin of cement mortars which had been exposed to the action of magnesium sulfate solution for a fairly long period sometimes appeared quite free from calcium sulphoaluminate crystals, while gypsum (CaSO4) was present in large quantity. The white solid layer which formed at the surface of cube samples was gypsum. In the interior of the samples, where access of the solution had been much slower, both calcium sulphoaluminate and gypsum were present as white crystals.

The conversion of calcium hydroxide to gypsum and the combination of hydrated calcium aluminate and gypsum in the solution to form calcium sulphoaluminate respectively involved an increase in the volume of the reacting solid by a certain factor. These reactions resulted in the expansion and disruption of mortars and concretes attacked by sulfate solutions. Hence, it was found from experiments that the specimens soaked in magnesium sulfates experienced weight gain and expansion. Magnesium sulfate formed a hard dense skin on mortars and concrete due to the deposition of magnesium hydroxide in the pores and this tended on the cube samples. A mortar attacked by magnesium sulfate more often consists of hard granular particles.

The compressive strength of different strength of all the series at different ages is shown in Figure 2. Many researchers have reported that the appearance of the strength was slowed in the early curing period by adding pozzolanic material, such as fly ash, because the overall pozzolanic reaction by a large amount within a few weeks (Targan, S. et al., 2003). From all results acquired, it was obvious that the concretes stored in MgSO4 solution at room temperature show a lower strength compared water-stored samples up to 90 days and not much compressive strength change was observed. Fly ash series observed that the compressive strength at 28 days of concretes were lower than the control samples. However, it was shown the higher compressive strength compared to control samples after 28 days up to 90 days and still lower compared to water-stored samples. Roughly, the strength of all mixtures were continuously increased up to 90 days except for 15% ash added, which strength decreased at 56 days but gains later up to 90 days. It is learnt that OPC hardened not through drying but through chemical reaction in among particle cement and air which known as hydration process.

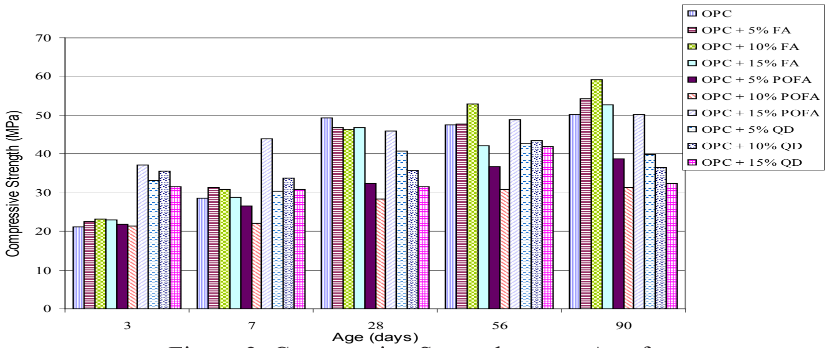

Figure 2: Compressive Strength versus Age for All Samples Immersed in MgSO4 Solution

POFA shows that at all ages, the compressive strengths of the concrete cubes decrease as the percentage of ash increases for 5% and 10% ash added. While, for 15% ash added, the compressive strength developement was higher in all ages. There was a continuously strength increase for all the mixtures with exposure age. Overall, all the mixtures in this series were lower in compressive strength compared to conctrol samples. Previous research has shown that POFA's use successor substance as cement will cause strength early concrete that lower achieve. This is because calcium content has diminished in amount of material cement in use concrete mixture caused by chemical composition over higher POFA content silica over calcium content. This resulted calcium hydroxide reaction Ca(OH)2 has been slow. Yet, at later ages, concrete compressive strength of concrete containing POFA increases. This is because calcium hydroxide and reaction cement hydration react with silica from POFA cementitious and produce gel C-S-H.

Quarry dust series shows that the compressive strength of concrete containing 5%, 10%, and 15% quarry dust decrease at 7 days, strength gains later at 28 days, and finally decrease again up to 90 days. The first decrease in compressive strength of concrete with quarry dust can attributed to the acceleration of sulphate attack which leads to the formation of much products from the reaction of magnesium sulphate with cement hydrates. Compared to reference concrete, concrete with quarry dust had higher strength at early ages but had lower strength after 28 days up to 90 days of concrete. The increase in compressive strength of concretes may be attributed to type of reaction. The continuous hydration of unhydrated cement components to form more hydration products in addition to the reaction of pozzolans with the liberated lime to form more C-S-H leading to increasing compressive strength and reaction of sulphate ions with hydrated cement components to form gypsum and ettringite.

Weight change was determined for all the samples stored in sulphate solution at room temperature. The results of weight change with exposure time for the various mixes are shown in Figure 3. There was a continuously weight increase for all the mixtures with exposure age with different extents except concrete containing 10% PFA and 5% QD at the 7 days of concrete. For control specimens, the results showed weight change of specimens decrease at 28 days of age but showed a continuous increase up to 90 days. From the data that has been shown, all concrete expand over with the first day concrete age after immersed into sulphate solution.

All samples of concrete did not show any weight reduction until 90 days compared to the first day of concrete cubes. Concrete expansion percent is the same or not much difference with reference concrete. The mass gain during the immersion period up to 90 days could be attributed to a number of factors of sulphate solution, which include continued hydration of cement, formation of gypsum and increase in absorbed water in samples. It was relatively higher mass gains occured with the concretes PFA10, POFA5, QD5, and QD10. For PFA5, PFA15, and QD15, concrete expansion percent was lower compared to reference concrete up to 90 days.

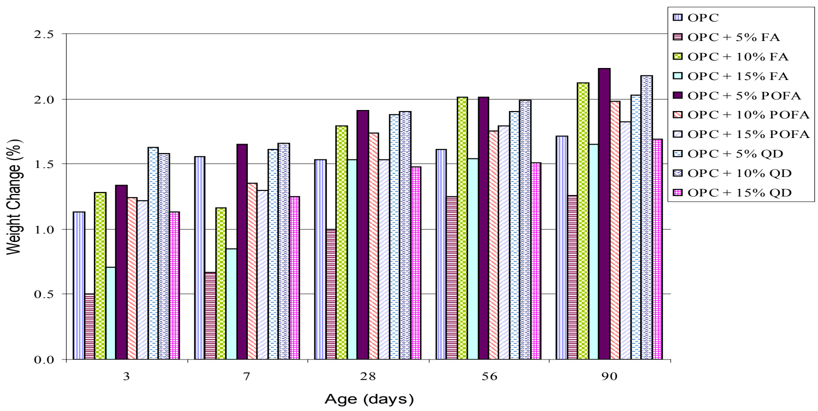

Figure 3: Percentage Weight Change versus Age for All Samples

It is obvious that the use of weight loss to predict the sulphate resistance is not an accurate method. A nearly similar was observed in the change of relative compressive strength for different series of all mixes with curing age. All the specimens showed a continuous increase of in compressive strength up to 90 days. Usually, sulphate attack causes mass loss by damaging Portland cement paste after a certain exposure time. In this project, the exposure time may not have been long enough to show the mass loss phenomenon for Portland cement paste. MgSO4 solution on the other hand does not produce a highly expansive reaction but significantly damages the resulting in a reduced compressive strength.

The length change of the specimens was measured using a digital vernier calliper. The first reading was taken 24 hr from the time the cement, aggregates, and water were mixed together. The average expansion value of three specimens was reported by getting the average volume of the specimens. From Figure 4, all concrete found experiencing increase to the length of concrete. See to the increased stated volume, conventional cocrete was expansion higher that is as many as 1.45% to 3 days of concrete age and expansion percent increasingly decline until to age 90 days which hit 0.5%.

It differs to the concrete containing fly ash, first of all percentages of volume expansion decrease at early days, volume gains later at 28 days, volume decrease again, and finally percentages of volume expansion increase up to 90 days except POFA15. The highest volume increase percent for concrete containing fly ash is at 56 days which is POFA15 as many as 2.44%. While, for the concrete with POFA cementitious, concrete volume percent change obtain stay in case out of phase. The highest percentage of concrete volume is very different over highest percentage value by reference concrete. The highest percentage volume increased for concrete with POFA is about 2.43% while the lowest is as many as 0.35%. Both percentages are the average percentage volume for POFA15 which were at 90 and 3 days respectively. However, concrete expansion which contains QD is a relatively small over other pozzolanic concrete in this study. Expansion percent lowest volume is QD15 which is 0.23% to 90 days. Highest percent is as much as 1.87% for QD5 to 28 days.

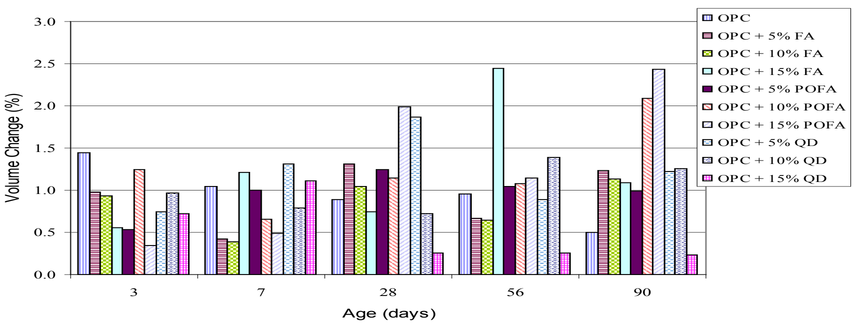

Figure 4: Percentage Volume Change versus Age for All Samples

6 CONCLUSIONS

The following conclusions can be drawn from this investigation:-

1. Concrete contains local pozzolan gave higher resistance against magnesium sulfate attack compared to conventional OPC concrete.
2. For each series, fly ash with 10% replacement, POFA with 15% replacement and 5% replacement of QD gave the highest resistence in each series.
3. Series of fly ash gave the best resistance followed by POFA series and lastly QD series.

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