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The effect of acidic environment on the concrete utilizing palm oil fuel ash

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Abstract. High-performance concrete (HPC) is widely used in construction projects, however, it has a large carbon footprint during the manufacturing of cement. Malaysia is one of the largest palm oil producers, and in the extraction of palm oil, it produces a lot of waste by-product, one of which is palm oil fuel ash (POFA) that would put an impact on the environment. Recently, there are many research studies revealed that POFA has a pozzolanic characteristics which can partially replace the cement to produce an eco-friendly concrete. This study illustrates the compressive strength and durability of concrete utilizing micro POFA. POFA, which was obtained from a local source, was sent for treatment and grinding procedure to remove excess carbon content and obtain 45 μ m size. 0%, 10%, 20% and 30% of micro POFA were used to replace the Portland cement in the used mixes. After curing age of 28 days, compressive strength and acid resistance of concrete cubes were investigated. Two different acids, hydrochloric acid (HCL) and sulfuric acid (H₂SO₄) were used to provide different aggressive environments for the concrete cubes. Two different optimum replacement percentages of POFA regarding to the acid resistance were determined by using the compressive strength loss, total mass loss and the visual inspection before and after the submersion. As two different acids were used, the rates of two acid attack on the concrete were studied in terms of mass loss throughout the submersion period. The results showed that the replacement percentage of 20% had the highest compressive strength in 28 days compressive strength test. Besides that, in terms of durability, even though the HPC utilizing 20% of replacement percentage of POFA did not have the highest acid resistance to the acid attack, however it had the highest compressive strength after the submersion in two different acids. This research showed that the replacement of micro POFA in HPC, possessed a significant improvement in terms of compressive strength and acid resistance.

1. Introduction

Ordinary Portland Cement (OPC) is the main material used when producing concrete and mortar, however it is also one of the biggest contributors in global carbon emission, which is 5-8% of the global carbon emission [1]. The global carbon emission can be foreseen to be increased in the next few years, due to the high demand of cement in developing countries [2] [3]. However, due to the environmental impact of cement, some developed countries have implemented the sustainable design and construction. Besides, few developed countries have begun the low carbon policies and regulations, such as Kyoto Protocol which aimed to reach a carbon emission reduction target [4].

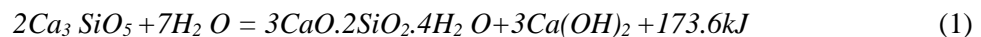
In order to minimize the environmental impact of cement, the researchers have found that cement can be replaced by recycled materials or by-products [5]. One of these materials is palm oil fuel ash (POFA), which is an industrial waste by-products of the palm oil production. Palm oil fuel ash contains silica and alumina when comparing the chemical composition with the cement. With the replacement of palm oil



fuel ash, it can help to increase the bonding between the concrete paste and aggregates, thus increase the strength and durability.

1.1 Portland Cement and Palm Oil Fuel Ash

The chemical composition of the materials which can replace the cement, should have a pozzolanic content, which is silicate-based content. In the cement hydration equation, silica will react with water to combine with calcium (lime based content) to form a bonding between the aggregates and the lime [6], and produces $3\text{CaO} \cdot 2\text{SiO}_2 \cdot 4\text{H}_2\text{O}$, which is also known as C-S-H, and C-S-H has played an important role in providing the concrete strength.



By comparing the chemical composition of palm oil fuel ash and cement, it can be found that palm oil fuel ash has a high content of silica, which can be used to replace cement. The palm oil fuel ash is a highly pozzolanic supplementary cementing material if compared its own chemical composition in accordance with ASTM C618 [7] [8]. Table 1 shows the chemical composition comparison of Cement and POFA.

Table 1: Chemical Composition of Cement and POFA [7]

Element	Mass Content (%)	
	Cement	POFA
Magnesium Oxide (MgO)	2.402	1.211
Aluminum Oxide (Al_2O_3)	3.443	3.153
Silicon Dioxide (SiO_2)	15.713	79.306
Phosphorus Oxide (P_2O_5)	0.388	2.322
Sulfur Trioxide (SO_3)	3.773	0.451
Potassium Oxide (K_2O)	0.265	3.233
Calcium Oxide (CaO)	70.428	2.793
Titanium Dioxide (TiO_2)	0.110	0.235
Manganese Oxide (MnO)	0.086	0.072
Iron Oxide (Fe_2O_3)	3.343	7.122
Copper Oxide (CuO)	0.011	0.016
Zinc Oxide (ZnO)	0.001	0.028
Rubidium Oxide (Rb_2O)	0.004	0.012
Zirconium Dioxide (ZrO_2)	0.008	0.044
Strontium Oxide (SrO)	0.025	-

1.2 Effect of Fineness of Palm Oil Fuel Ash

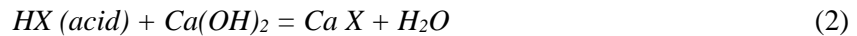
Palm oil fuel ash with different fineness has brought the effect on the compressive strength of the concrete [9]. Three different fineness of palm oil fuel ash were researched [9], which are treated palm oil fuel ash, fine treated palm oil fuel ash and ultra-fine treated palm oil fuel ash. The result of the research has shown that the higher fineness of the treated palm oil fuel ash has the lower percentage of unburned carbon, which will influence the strength of concrete [10].

Without appropriate treatment, palm oil fuel ash can only be able to provide minted pozzolanic potential due to its large-sized particles and higher content of residual carbon, which is organic content [11]. The recommended treatments to the palm oil fuel ash that are used as partial cement replacement materials, are heat treatment and fine grinding, to enhance the pozzolanic potential of palm oil fuel ash [9].

1.3 Acid Attack on Concrete

In the real life, concrete may be exposed to different environments, including aggressive environments, which may degrade the concrete. When concrete loses its strength, it may cause structure failure, which is needed to be avoided. The possible aggressive environments that may contact with concrete are acid attack, fire, chloride attack etc. In this research, acid attack is only considered. When concrete is contacted with acid, acid will react with the calcium hydroxide, which is a product in the hydration, to form the calcium salt ($\text{Ca}(\text{OH})_2$), which is mostly soluble in water. The calcium salt will escape from

concrete, and weaken the concrete paste's structure. The process in which concrete loses its strength is called deterioration. For the insoluble salt which is a minor product in the acid reaction, these salts will remain in concrete layers and form a thin protection, which can slow down the deterioration process. Eq. 2 shows the acid attack on concrete.



1.3.1 Hydrochloric Acid

Hydrochloric acid is widely used in the industrial world. It can be used to remove impurities in steel, production of batteries, photoflash bulbs and also help in processing sugar [12]. In industry, concrete is used for the storage of hydrochloric acid for cost saving, which is harmful to concrete. Besides, during the storage of hydrochloric acid, there is acidic fume released, which will damage the nearby structure, such as concrete floor or column.

Hydrochloric acid attack also occurs in chimneys, which are used to provide ventilation for gases or smoke to the outside atmosphere. Adding to that, when the hydrochloric acid fume—which is released from the industry—is mixed with rain, it will form acid rain. The acid rain will cause acid attack to the exposed concrete structure, such as concrete buildings, floors and bridges. If concrete structures fail to support their design loads due to the acid attack, it will cause structural collapse, which will lead to injuries and death.

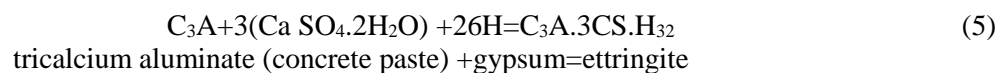
1.3.2 Sulfuric Acid

Sulfuric acid, H_2SO_4 is a strong oxidizing agent. When this acid reacts with concrete, calcium sulfates will be produced as shown in Eq. 3.



Calcium sulfate, $CaSO_4$, which is also known as gypsum, will dissolve in water and form a sulfate solution. In the acid attack, sulfuric acid attack can be said that it is very damaging to concrete, as if acid attack and sulfate attack combined at the same time. Acid attack is caused by the hydrogen ions, while sulfate attack is caused by the sulfate ions.

Sulfate attack is formed when there is a presence of the sulfates of calcium, sodium, potassium or magnesium. When sulfuric acid contacts with concrete, the calcium salt will be escaped from the concrete in the early stage. Solid salts usually do not attack concrete, but if the sulfate salt is present in solution, it will react with the concrete paste (Tricalcium aluminate, one of the products in concrete) to form the ettringite. Ettringite is a crystal form salt, which will fill in the void in concrete and cause expansion and disruption of concrete. The expansion and disruption of concrete will cause cracking and loss of bond between the concrete paste and aggregates.



Despite the use of sulfuric acid in the industry for manufacturing, there are other sulfuric acid sources which have a higher chance to contact with concrete. Sulfuric acid is commonly used in agriculture, as sulfuric acid can be used as a fertilizer. With the use of the fertilizer, sulfuric acid may be washed down to rivers during raining, and contacts with concrete structures, such as bridges and sewer pipes. On the other side, there is sulfate-based soil present in nature, such as peat soil. Besides that, with the use of sulfate-based fertilizers in agriculture, the particular soil will contain a certain content of sulfate. When the soil microbes and the water react with the sulfate content in the soil or in the water, it will form sulfuric acid, which will cause acid attack to concrete under soil or water. The conversion from sulfate to sulfuric acid usually takes a few weeks and will be more efficient in a warm environment. Besides that,

the sulfuric acid attack is happened to the steam railway tunnel due to the steam released from steam locomotives.

2. Materials and Methodology

OPC and POFA were used as binders, sand and quarry dust were used as fine aggregates while the crushed granite was used as coarse aggregates. POFA was purchased from local Palm oil mill. POFA was sent for treatment, followed by grinding to obtain the 45 μ m. Table 2 shows the mix proportions of concrete.

Table 2: Mixture Proportion of High-Performance Concrete

Material	Concrete Mixture			
POFA %	0	10	20	30
Cement	588	529.2	470.4	411.6
POFA	-	58.8	117.6	176.4
Coarse Aggregates	1093	1093	1093	1093
Sand	268	268	268	268
Quarry Dust	268	268	268	268
Water	183	183	183	183
SP	0.2	0.2	0.2	0.2

Four different concrete mixtures were prepared according to ACI 211.4R standards [13]. Each batch with a size of 100 mm \times 100 mm \times 100 mm, were having 9 cubes, with total specimens of 36 cubes. Slump test was conducted according to ASTM C143 [14]. After 24 hrs of casting, all cube specimens were demoulded and cured in water for 28 days. In each batch, 3 cubes were used for 28 days compressive test, 3 cubes were submerged in HCL and the remaining 3 cubes were submerged in H₂SO₄. The submersion period was 75 days or 1800 hrs, according to ASTM C267 [15]. In the meanwhile, the mass of concrete cubes was taken out and rinsed for every 7 days to measure the mass loss for rate of acid attack used. After 75 days, the cubes were sent for compressive strength test to determine the strength loss. Before the compressive strength test, concrete cubes were under a visual inspection to compare the physical changes before and after the submersion.

3. Result and Discussion

3.1 Slump Test

The workability of concrete can be determined from the slump test, which was conducted for every casting batch. Every batch of concrete has a constant water-cement ratio of 0.308 and the superplasticizer dosage of 0.2% of cement weight. Table 3 shows slump test results of each batch to compare the effect of palm oil fuel ash replacement on the workability.

Table 3: Slump Test Results

POFA replacement percentage (%)	slump height (mm)
0	100
10	88
20	54
30	33

From Table 3, it can be seen that the slump height decreases when the replacement percentage of palm oil fuel ash increases. It can show that the higher the palm oil fuel ash replacement percentage, the higher the water demand to maintain the same workability. It is due to the high porosity of palm oil fuel ash

and the specific surface area (surface fineness), which will cause the palm oil fuel ash to have a higher water absorption than cement. As the palm oil fuel ash absorbs the water, there is less free water in the concrete mix, thus causing the workability of the concrete drops.

3.2 Compressive Strength Test

The 28 days compressive strength test results of concrete were required for each batch. In compressive strength test, three 100 mm × 100 mm × 100 mm concrete cubes for every batch were tested to obtain the average compressive strength of each concrete mix.

Table 4: 28 Days Compressive Strength of Concrete

POFA replacement percentage (%)	compressive strength (28 days) (MPa)			
	1	2	3	Average
0	68.23	63.8	66.79	66.27
10	71.39	73.37	69.88	71.55
20	81.95	85.35	79.15	82.15
30	66.45	71.69	68.8	68.98

By increasing the palm oil fuel ash replacement percentage, it can be seen from Table 4 that the compressive strength of concrete is increasing due to the chemical composition change. From equation 1, it has shown that C-S-H has an important role by providing the concrete strength. However, from Table 1, cement has a lower composition in silica, which roles as a limiting reagent, thus it will limit the production of C-S-H. Limiting reagent is usually meant as the lowest amount of reagent in a chemical reaction, as it will determine the amount of products that will be formed. In control concrete, the silica content is significantly lower. However, with the palm oil fuel ash replacement, which has a high content in silica and low content in calcium, it will increase the amount of silica content in the concrete mixing. It means that the limiting reagent, which is silica content is increasing, thus help in producing the C-S-H.

However, from Table 4, the concrete which contains the 30% palm oil fuel ash replacement has a lower compressive strength than the 20%. It is due to the significant decrease in calcium content, and it will make the calcium content as the limiting reagent. As calcium is limited to produce the C-S-H and calcium salt to form a complete hydration process, the formation of C-S-H will be decreased, thus the strength is decreasing. Therefore, the optimum replacement percentage of palm oil fuel ash is 20%, as silica and calcium can almost fully function in producing the C-S-H. In terms of improving the 28 days compressive strength, 20% of the palm oil fuel ash as the partial cement replacement is the optimum replacement percentage.

3.3 Compressive Strength Loss after Deterioration

The comparison of compressive strength of concrete cubes are used to determine the acid resistance of the concrete cubes utilizing the palm oil fuel ash replacement percentage.

Table 5: Compressive Strength of Concrete Cubes after Immersed in HCL and H₂SO₄

POFA %	Compressive Strength After Submerged in (MPa)		Strength loss (%)	
	HCL	H ₂ SO ₄	HCL	H ₂ SO ₄
0	45.33	37.99	31.64	42.68
10	58.17	47.67	18.69	33.38
20	66.34	55.24	19.24	32.75
30	53.59	37.97	22.31	44.96

From Table 5, it shows that the strength loss decreases when the replacement percentage of palm oil fuel ash is increasing. Addition of palm oil fuel ash can help in increasing the production of C-S-H, and reducing the formation of calcium salt. It is because when the C-S-H production increases, the calcium left over will be decreased, which will lead to less formation of calcium salt. As less calcium salt is produced, less void will be formed and thus helps in reducing the strength loss. On top of that, the presence of palm oil fuel ash will cause the pozzolanic reaction which make the Ca(OH)₂ to form secondary C-S-H to fill in the existing void, thus making the concrete denser [16].

However, with the excessive use of palm oil fuel ash, the compressive strength of concrete starts to be reduced. When there is excessive use of palm oil fuel ash as partial cement replacement, it lowers the calcium content significantly, which will lead to lesser hydration process. As hydration process is limited, it causes less formation of C-S-H and calcium salt, thus slow down the hydration rate, and causing hydrated C-S-H. When the concrete cubes are immersed in the acid, the acid will attack the hydrated C-S-H paste, and it will be broken down and producing large amount of calcium salt, and lead to less dense concrete, thus increasing the compressive strength loss [16].

In terms of compressive strength loss, there are two optimum replacement percentages regarding the two different acids due to the different mechanism of the acids. From Table 5, 10% of palm oil fuel ash is the optimum replacement percentage to resist the HCL acid attack; while 20% of palm oil fuel ash is the optimum replacement percentage in order to resist the H₂SO₄ acid attack.

3.4 Mass Loss after Deterioration

Throughout the process of deterioration, the mass of the concrete cubes were measured to determine the mass loss. Mass loss of concrete cubes can be used to determine the acid resistance of the concrete and rate of acid attack.

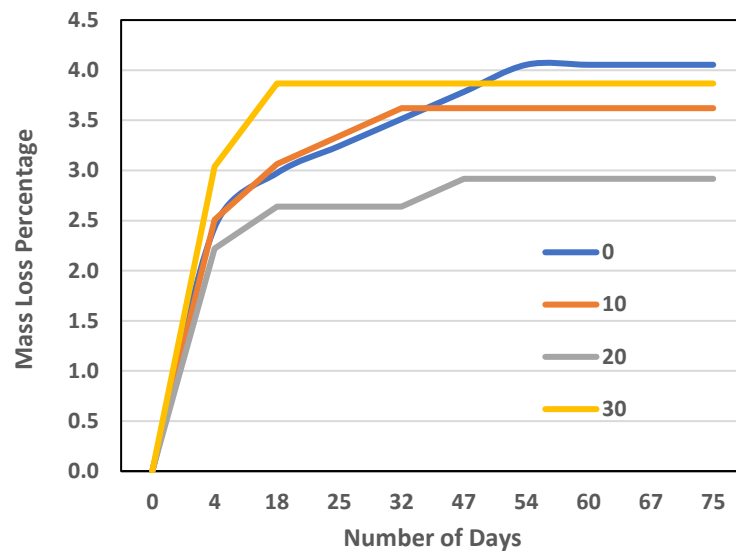


Figure 1 Cumulative Mass Loss of Concrete Cubes Immersed in HCL

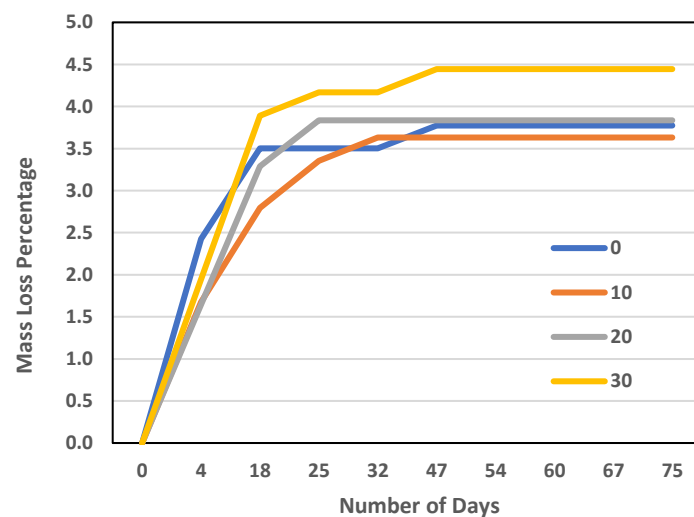


Figure 2 Cumulative Mass Loss of Concrete Cubes Immersed in H₂SO₄

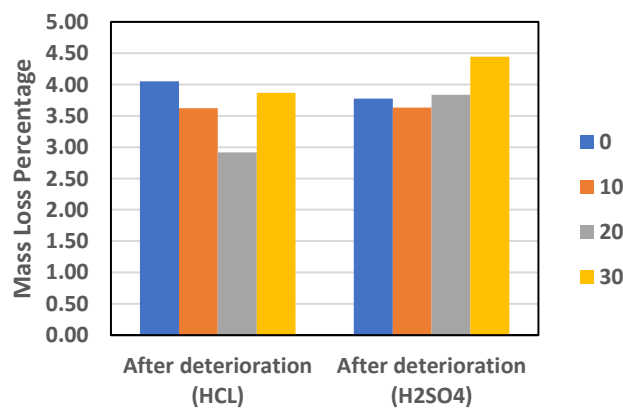


Figure 3 Total Mass Loss of Concrete Cubes after Deterioration Process

From Figures 1 and 2, the masses of the concrete cubes drop greatly at the first few days. However, after few weeks, the mass loss rate is decreasing as most of the salt are reacted with the acid. Besides that,

the insoluble salt formed in acid attack reaction are attached on the concrete layer and forms a thin layer to slow down the deterioration rate. From the mass loss of the concrete cubes, it can show the rate of acid attack on the concrete. It can be determined that the hydrochloric acid and sulfuric acid are strong acids, which will form a very fast reaction with concrete.

Figure 3 shows that the palm oil fuel ash can help in improving the acid resistance as the mass loss drops with the addition of palm oil fuel ash as partial cement replacement. It is due to that less salt are formed in hydration process, thus less salt is escaped from the concrete cubes. However, the mass loss of the concrete cube increases with the excessive use of palm oil fuel ash. As less hydration process occurred when it comes to excessive use of palm oil fuel ash due to the low content of calcium, hydrated C-S-H is formed greatly. When the hydrated C-S-H reacts with the acid, it will be broken down and produce large amount of calcium salt, thus causing the concrete to be less dense. Besides, this breaks the binding between aggregates thus lead to detachment of the particle which results in greater mass loss, which proceeds to strength loss [16].

In terms of mass loss, 20% palm oil fuel ash is the optimum replacement percentage to resist HCL acid attack while 10% palm oil fuel ash is the optimum replacement percentage to resist H_2SO_4 acid attack. From the results of the compressive strength loss and mass loss, the concrete cubes which are immersed in H_2SO_4 has the greater losses than the one immersed in HCL. It is because when the concrete cubes are facing the H_2SO_4 acid, they have to resist against acid attack and sulfuric attack. As the calcium salt reacts with H_2SO_4 , calcium sulphate salt will be produced and dissolve in water, which will form a sulphate solution, that is another aggressive environment to the concrete. The sulphate solution will react with the concrete paste to form ettringite, which will cause expansion and disruption of concrete. Besides that, it will cause more aggregates to lose bonding as concrete paste is disrupted, more detachment of the concrete paste, which will result in larger strength loss and mass loss.

3.5 Visual Inspection

Visual inspection can help to determine the performance of the concrete, as the concrete strength can be affected by many issues. Visual inspection was conducted after the concrete cubes were submerged in the hydrochloric acid and sulfuric acid. Table 6 shows the result of the visual inspection on the concrete before and after the acid exposure.

Table 6: Visual Inspection on the Concrete Before and After Acid Exposure


























Replacement Percentage of Palm Oil Fuel Ash	Surface contact with the bottom layer of container	Side Surface	Surface facing upwards
Concrete before submersion in acid			

Figure 4 Surface of Concrete Before Deterioration

Sulfuric Acid			
0	 <p>Figure 5 Bottom Surface of 0% Concrete (H_2SO_4)</p>	 <p>Figure 6 Side Surface of 0% Concrete (H_2SO_4)</p>	 <p>Figure 7 Top Surface of 0% Concrete (H_2SO_4)</p>
10	 <p>Figure 8 Bottom Surface of 10% Concrete (H_2SO_4)</p>	 <p>Figure 9 Side Surface of 10% Concrete (H_2SO_4)</p>	 <p>Figure 10 Top Surface of 10% Concrete (H_2SO_4)</p>
20	 <p>Figure 11 Bottom Surface of 20% Concrete (H_2SO_4)</p>	 <p>Figure 12 Side Surface of 20% Concrete (H_2SO_4)</p>	 <p>Figure 13 Top Surface of 20% Concrete (H_2SO_4)</p>
30	 <p>Figure 14 Bottom Surface of 30% Concrete (H_2SO_4)</p>	 <p>Figure 15 Side Surface of 30% Concrete (H_2SO_4)</p>	 <p>Figure 16 Top Surface of 30% Concrete (H_2SO_4)</p>

Hydrochloric Acid			
0			
	Figure 17 Bottom Surface of 0% Concrete (HCl)	Figure 18 Side Surface of 0% Concrete (HCl)	Figure 19 Top Surface of 0% Concrete (HCl)
10			
	Figure 20 Bottom Surface of 10% Concrete (HCl)	Figure 21 Side Surface of 10% Concrete (HCl)	Figure 22 Top Surface of 10% Concrete (HCl)
20			
	Figure 23 Bottom Surface of 20% Concrete (HCl)	Figure 24 Side Surface of 20% Concrete (HCl)	Figure 25 Top Surface of 20% Concrete (HCl)
30			
	Figure 26 Bottom Surface of 30% Concrete (HCl)	Figure 27 Side Surface of 30% Concrete (HCl)	Figure 28 Top Surface of 30% Concrete (HCl)

3.5.1 Visual Inspection on Concrete Submerged in H_2SO_4

Figure 4 shows concrete surface before submerging in the acid. From Figures 5 to 16 shown above, concretes that were submerged in sulfuric acid have a rust color stain and a milky color thin layer. Rust color indicates the areas with intense degradation [17], while milky color indicates the gypsum layer. In sulfuric acid attack, calcium sulfate is produced, and it is not soluble in water. The salts will remain in water and stick to the concrete surface due to settlement. From the side views of the concrete, they have a milky color and rust color on each side. In the deterioration process, when concretes sat in the container, the salt produced will sink to the bottom, and be stacked as more salt are produced. It will form a protective layer to the concrete to slow down the deterioration rate. Therefore, the sides of the concretes have a different color.

Besides, by observing the physical changes on the concretes that are submerged in the sulfuric acid, the aggregates of the concretes are exposed, which cause a rough concrete surface, and lead to lower the compressive strength. However, by comparing Figure 6 and Figure 12, the concrete with 0% of palm oil fuel ash has a more severe exposure of aggregates, while the concrete with 20% of palm oil fuel ash has the least severe of the aggregates' exposure. It shows that the concrete with 20% replacement of palm oil fuel ash has a higher resistance to sulfuric acid attack, as less concrete paste is detached.

3.5.2 Visual Inspection on Concrete Submerged in HCL

From the observation on Figures 17 to 28, the physical appearance of the concrete, which were exposed to the hydrochloric acid changes, as the colors of the concrete cubes went brownish and darker. Besides, the surface of the concretes became porous with a rust color inside. One can determine the severity of the concrete by observing the color changes. The concrete with 0% of palm oil fuel ash had a severe condition as its colors turned into a total dark brown color. It means that the concretes have lost more surface compound, which being damaged by the acid. By comparing Figure 19, Figure 22, Figure 25 and Figure 28, the concretes with 10% replacement percentage of palm oil fuel ash had a better resistance to the hydrochloric acid, as its color almost remain as the same, and it has less pores on the surface.

Besides, when observing the concretes, the surface of the concretes gave a sandy surface. It is due to the fact that the insoluble salt retains on the concrete surface. Chandra 1988 mentioned that when the calcium salt reacts with the hydrochloric acid, it forms soluble and insoluble salt. The insoluble salt will form a layer (hydroxide mixture) on the concrete and form a dark brown layer. It means the brownish the concrete is, the more salt is produced, thus lead to losing the strength as the concrete becomes less dense.

4. Conclusions

Based on the results obtained, it can show that the partial replacement of POFA as cement can help in strength and durability. In terms of workability, extra dosage of SP is needed when it comes to the higher replacement percentage of POFA because micro POFA has a higher water absorption. In terms of 28 days compressive strength, 20% is the optimum replacement percentage. However, when it comes to the acid resistance, different optimum replacement percentages were obtained regarding to different lab tests and different acidic environments. However, there are many issues which will affect the compressive strength loss, such as in-dent conditions and exposure of aggregates. As the physical changes of concretes after deterioration cannot be predicted and estimated, the optimum replacement percentage from strength loss can be used as a reference.

It is recommended to replace ordinary Portland cement with 20% of palm oil fuel ash. Although it has a slightly lower performance in mass loss and strength loss when it is facing different acids, but from the results, it can be seen that the concrete with 20% palm oil fuel ash has the highest compressive strength before and after the deterioration process.

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