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Seawater Exposure Effect on Fly Ash based Geopolymer Concrete with Inclusion of Steel Fiber

Zarina Yahya^{1,2,*}, Mohd Mustafa Al Bakri Abdullah¹, Leow Yee Jing², Long-Yuan Li³, and Rafiza Abd Razak^{1,2}

¹Center of Excellence Geopolymer and Green Technology, School of Materials Engineering, Universiti Malaysia Perlis (UniMAP), 01000, P.O. Box 77, D/A Pejabat Pos Besar, Kangar, Perlis, Malaysia

²Department of Civil Engineering Technology, Faculty of Engineering Technology, Universiti Malaysia Perlis (UniMAP), Sungai Chuchuh, 02100 Padang Besar, Perlis, Malaysia

³School of Engineering, University of Plymouth, Plymouth, PL4 8AA, UK

Email: * zarinayahya@unimap.edu.my

Abstract. Concrete is widely used in construction offshore such as concrete floating bridges and sea tank. This research is providing an alternative construction material to replace ordinary Portland cement (OPC) concrete known as geopolymer. The geopolymer concrete was produced by mixing fly ash with alkaline activator and 3% of steel fibre in order to improve the properties of fiber reinforced geopolymer concrete (FRGPC). The effects of aging period in term of strength, changes in weight and carbonation of FRGPC in seawater is investigated and compared with the fiber reinforced concrete (FROPC). The compressive strength obtained for FRGPC were higher than FROPC. The highest compressive strength obtained by FRGPC is 76.87 MPa at 28 days and 45.63 MPa at 28 days for FROPC concrete. The compressive strength was decreased as the period of immersing the concrete in seawater is increased. During the immersion process of both samples in seawater up to 120 days, the carbonation was not detected even though with the existence of steel fibres.

1. Introduction

Green concrete was developed rapidly in recent years in order to reduce carbon footprint especially carbon dioxide (CO₂) emission and limited landfill areas. By definition, green concrete can be defined as concrete that are produced using waste materials in order to decrease the natural resources depletion, environmental pollution as well as energy consumption [1]. Geopolymer has been introduced by Davidovits in 1978 where waste materials that rich in Silica (Si) and Aluminium (Al) was used as source materials. When these source materials activated with alkaline activator solution it possesses binder properties which is similar with ordinary Portland cement (OPC). The by-product materials such as silica fume, fly ash, rice husk ash, slag, red mud and natural minerals like kaolinite, albite, clays, feldspar and stilbite are the example of source materials [2-7]. However, slags, calcined clays and coal fly ashes are the common source materials used by the researchers for geopolymer [8]. Hardened binder is produced when the source materials were dissolved in an activating solution polymerizes into molecular chains and networks.

Geopolymer concrete plays an important role in producing a green environment. A number of 3.6 billion tons of cement have been produced in 2011 showed that the production of cement has increased



year by year [9]. Cement production caused the liberation of CO₂ to surrounding due to burning of fossil fuels and the calcination of limestone. Therefore, the emission of CO₂ should be reduced by using the geopolymer concrete instead of OPC. This would help in turning down the global thermostat.

The inherent weakness of plain concrete is caused by the presence of micro-cracks in the concrete aggregate interface [10]. The addition of fibres in the mix can increase the strength of concrete. This is due to the loads transferred by the fibres at the internal micro-cracks. Therefore, adding the fibres which are short, discontinuous and discrete fine fibres of specific geometry dispersing randomly in the conventional concrete form a new composite material to a concrete. The function of fibres is like an aggregate but it has a totally different shape compared to aggregates which is rounded smooth. The fibres act as an interlocking agent assist to interlock and fill the voids between aggregates in the concrete. This will cause the workability of the concrete decreases as the mix is more cohesive and less prone to segregation [11]. Fibres can reduce the development of cracks and transform the properties of concrete with low tensile and impact resistance into a strong composite that has an excellent resistant to crack, good ductility and post-cracking behavior prior to failure. Based on the research by Hake et al. [12], the compressive strength of geopolymer concrete (GPC) increased when the percentage of steel fibre increases from 1% to 3%. Besides, compressive strength and flexural strength are enhanced when the steel fibres are added in alkali activated concrete. An optimum content of 3% of steel fibres is added and the optimum compressive strength is gained [11]. Conversely, Bernal et al. [13] investigated the effect of steel fibre on slag based geopolymer and the result showed reduction in compressive strength but enhanced the flexural strength and splitting tensile. The inclusion of steel fiber also improves the durability performance of alkali activated slag concrete in term of water absorption, capillary and water resistance penetration.

Concrete structures exposed to marine environment are highly vulnerable to corrosion due to chloride penetration and carbonation which compromise the durability of the concrete [14]. Moreover, the existence of various ions such as sulphates and magnesium in seawater contributed to the deterioration of concrete [15, 16]. Geopolymer exposed to seawater is subjected to high concentration of chlorides, sulphates and magnesium ions where it can cause several mechanisms such as crystallization of expansive salts, precipitation of insoluble composites or ionic attacks [17]. It was found that the concentration of ions in typical seawater is about 19,090 ppm of chloride and 2233 ppm of sulphate [18]. Previous researcher has found that during laboratories studies, the performance of geopolymer concrete (GPC) in term of chloride penetration especially chloride diffusion was relatively low compared to OPC concrete [19, 20]. Astutiningsih et al. [21], investigated the performance of fly ash and metakaolin based geopolymer concrete on seawater exposure for 7, 28, 56 and 90 days. It was found that the strength of fly ash geopolymer concrete decrease 20% during 56 days of exposure but did not decreased further until 90 days. Meanwhile, kaolin geopolymer concrete showed better performance due to the inexistence of calcium content. However, for controlled sample (OPC concrete) which consists of high amount of calcium showed major decrement in strength after exposure in seawater. Besides that, permeability also plays an important role in determining the durability of geopolymer in marine environment where denser geopolymer samples will be difficult for aggressive ions to penetrate through the pores. Frequently, previous research works more focused on one type of chemical exposure such as acid, sulphate and magnesium attack.

Vijai et al. [22] conducted a study on geopolymer concrete composite (GCC) that consists of 90% fly ash, 10% OPC and steel fibers. The steel fibers of 0.25% to 0.75% by volume in GCC enhanced the compressive strength, split tensile strength and flexural strength with 0.75% steel fiber contents contributed to optimum performance. The performance of fiber reinforced oil palm shell geopolymer concrete (FROPSGPC) prepared with mixture of slag, palm oil fuel ash (POFA) and 0.5% steel fibers was reported by Islam et al. [23]. The mechanical properties such as splitting tensile and flexural strength increased by about 19%-38% and 13%-44% compared to samples without addition of steel fibers.

This paper reports on the performance of fiber reinforced fly ash based geopolymer concrete (FRGPC) with 3% inclusion of wool steel fibers. The results are compared with fiber reinforced OPC concrete (FROPC), after both of samples were exposed in seawater up to 120 days. The performance of both types of concretes due chloride ingress from the seawater were measures by spraying phenolphthalein solution.

2. Experimental Methods

2.1. Materials

Fly ash was obtained from Cement Industries of Malaysia Berhad (CIMA), Perlis, Malaysia and it was categorized in class F according to ASTM C618-12 with the total content of silicon oxide, aluminum oxide and iron oxides more than 70%. This fly ash was used to produce fiber reinforced geopolymer concrete (FRGPC).

The Portland cement used to produce FROPC as a control concrete in this research is ASTM Type I Portland cement. Table 1 list the chemical composition of fly ash and OPC as determined by X-ray Fluorescence (XRF).

Table 1. Chemical composition of fly ash and OPC.

Composition	Fly Ash Weight (%)	OPC Weight (%)
SiO ₂	55.9	20.99
Al ₂ O ₃	27.8	6.19
CaO	3.95	65.96
Fe ₂ O ₃	7.09	3.86
TiO ₂	2.25	-
K ₂ O	1.55	0.60
SrO	0.37	-
ZrO ₂	0.13	-
RuO ₂	0.24	-

A technical grade of waterglass or sodium silicate solution was purchased from South Pacific Chemical Industries Sdn. Bhd. (SPCI), Malaysia. The waterglass consists of 30.1% SiO₂, 9.4% Na₂O and 60.5% H₂O (modulus SiO₂/Na₂O = 3.2), with the specific gravity of 1.4 g/cm³ and viscosity of 0.4 Pas (at 20 °C).

Sodium hydroxide (NaOH) powder had 99% purity and was made in Taiwan under the brand name of Formosoda-P. The NaOH solution of 12 Molar is prepared by diluting NaOH powder with distilled water and allowed to cool down to room temperature before it was used. Activator solution was prepared by mixing waterglass with NaOH solution. The fly ash/alkaline activator ratio was fixed at 2.0 and sodium silicate/sodium hydroxide ratio was fixed at 2.5.

River sand was used as fine aggregates with size not more than 4.75mm. Coarse aggregates of maximum sizes 20mm with specific gravity 2.5 and water absorption of 0.17% were used.

High quality of low carbon steel with straight end is the steel fibres used in this research. The dimension of steel fibres with the diameter of 0.13mm and the length of 5mm.

2.2. Preparation of Samples

The percentage of aggregates used to produce FRGPC is 70% from the total concrete mixture. The proportion of coarse aggregates and fine aggregate are 60% and 40% of the total mass of aggregates used. The percentage of steel fibres in the FRGPC is 3% from the total weight of the concrete which is the optimum content percentage with the highest strength gained based on previous study by Meor et al. [11]. The mix proportion and quantity of fibre steels content in each mix is shown in Table 2.

Table 2. Details of mixture proportions for FRGPC and FROPC.

Mix No.	FRGPC	FROPC
Fly Ash (kg/m ³)	8.2	-
Cement (kg/m ³)	-	8.2
Coarse aggregates (kg/m ³)	16.1	16.1
Fine aggregates (kg/m ³)	10.8	10.8
NaOH solution (kg/m ³)	0.9	-
Na ₂ SiO ₃ solution (kg/m ³)	2.4	-
Steel Fiber (kg/m ³)	1.2	1.2
Water (kg/m ³)	-	3.3

Fly ash and alkaline activator solution were mixed thoroughly using a mixer in a dry state. Then, coarse aggregates, fine aggregates and steel fibres were added into the mix until it is in homogeneous state. After the concrete had been thoroughly mixed, the fresh concrete was poured into the mold with dimension of 100mm×100mm×100mm. Each cube was casted in three layers. The fresh concrete was poured into the molds layer by layer for three layers and compacted it using the rod by dropping the rod to the fresh concrete for at least 25 times. Besides, at least 10 times was tapped on the side for each layer. The sample was vibrated by using vibrating table. All the samples were finished by using trowel to smooth the exposed surface of the concrete. The control samples (FROPC) also were casted with the same mix design as FRGPC.

After casting, the samples were preserved in the lab at room temperature for one day before being de-molded. Then, the samples were removed from the molds and cure at room temperature for 3 days. After 3 days of curing, the samples (FRGPC and FROPC) were situated in the water tank which filled with seawater and immersed for 28, 60, 90 and 120 days.

2.3. Testing

The compressive strength for FRGPC and FROPC samples were determined according to British Standard BS EN 12390-3 [24]. The compressive strength was measured when the concrete samples was crushed by using Universal Testing Machine (UTM). For each test, which are at 28th, 60th, 90th and 120th days, three concrete cubes were tested in order to obtain the average compressive strength values.

Carbonation depth in hardened concrete is determined by using phenolphthalein method with accordance to British Standard BS EN 14630 [25]. The test cube samples were taken out from the seawater tank at 28th, 60th, 90th and 120th day and split it by using concrete cutter. After the splitting of concrete sample was done, the freshly split concrete sample was cleaned and the phenolphthalein pH indicator was sprayed on the concrete sample. Then, the changes of colour on the concrete was observed. A purple colour is obtained on the concrete sample for the non-carbonated part of the samples as it is highly alkaline. While, no changes of colour is occurred for the carbonated part of the concrete samples as the low alkalinity of the concrete. The depth of the colourless phenolphthalein region was determined by measuring the depth of total six points which are three points perpendicular to the two edges of the split face of fresh concrete. The depth was measured immediately after spraying the phenolphthalein indicator. The average depth of the region of colourless surface was calculated.

3. Results and Discussion

3.1. Compressive Strength

Figure 1 showed the compressive strength of FRGPC and FROPC after being exposed to seawater for 28 days until 120 days. As it was expected, the compressive strength values for both samples demonstrated a linear decreasing trend as the exposure period increased. The highest compressive strength of 76.87MPa is obtained at 28 days whereas the lowest compressive strength obtained is

51.63MPa at 120 days for FRGPC samples. The trend for compressive strength of FROPC samples is almost identical with FRGPC. The highest compressive strength for FROPC is 45.63MPa at 28 days whereas the lowest compressive strength is 40.1MPa. The strength decrement for FROPC samples was almost constant after 60 days of exposure periods. Generally, the hydration process of OPC rely on lime (calcium) for strength properties where calcium silicate hydrate (CSH) and calcium hydroxide ($\text{Ca}(\text{OH})_2$) as the main hydration product. However, due to the depletion of CSH in FROPC hence caused deterioration of strength.

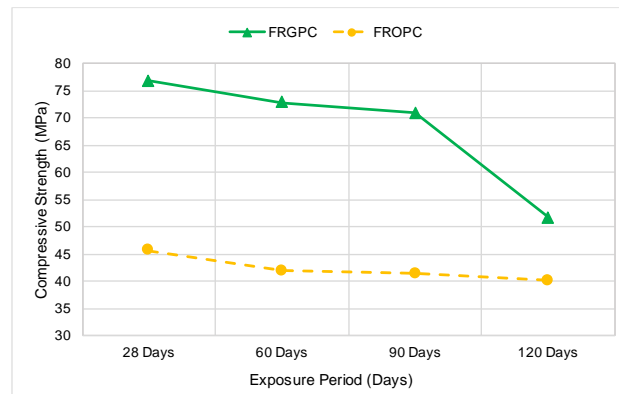


Figure 1. Compressive strength of FRGPC and FROPC.

The results obtained is accordance with the past research, which the compressive strength of geopolymer concrete is decreasing as the immersion time in seawater is increasing. In fact, the compressive strength of geopolymer concrete at 28 day was able to achieve highest compressive strength. This is due to low porosity of geopolymer concrete that resulting to lesser amount of sulfate ions can be ingress into geopolymer concrete [26]. Sulfate ions found in seawater will cause deterioration and loss in strength of concrete. Loss of cohesion of geopolymer concrete sample is caused when it is exposed to an external source of sulfate deteriorated by expansive reactions with magnesium hydroxide ($\text{Mg}(\text{OH})_2$) and calcium sulfate (CaSO_4).

FRGPC has higher compressive strength than FROPC and this may be due to the lower calcium content in the fly ash that effect the compressive strength of concrete. While, FROPC sample contain more calcium content due to the presence of Calcium Silicate Hydrate gel (C-S-H). The presence of calcium compounds will affect the ability of concrete to resist the chemical agents such as chloride ion when exposed to seawater [27]. Ingression of sulphate ions or chloride ions is not allowed in geopolymer concrete as geopolymer concrete does not have transition zone [28].

3.2. Changes in Weight

Figure 2 displayed the change in weight for both types of samples which are subjected to continuous immersion in seawater. Variation in weight of concrete is calculated with difference in initial weight and weight after period of exposure to seawater. There was no reduction in the weight of all the samples, but weight of samples showed small increment due to the exposed liquid which is the seawater was absorbed by the concrete.

According to Figure 2, the lowest variation of change in weight for FRGPC concrete is 0.4% at 28 days. While, the highest variation of change in weight for FRGPC is 1.23% at 120 days. For FROPC which is the control concrete is same with FRGPC has the lowest variation of change in weight of 2.11% at 28 days. While, the highest variation of change in weight is 4.22% at 120 days for FROPC. The trend line pattern for FRGPC is almost identical to that FROPC which is increasing trend as the age in seawater is increasing. However, the trend line for FROPC is above that of FRGPC. Therefore, the test results show that the variation of change in weight of FROPC is generally higher than that of FRGPC.

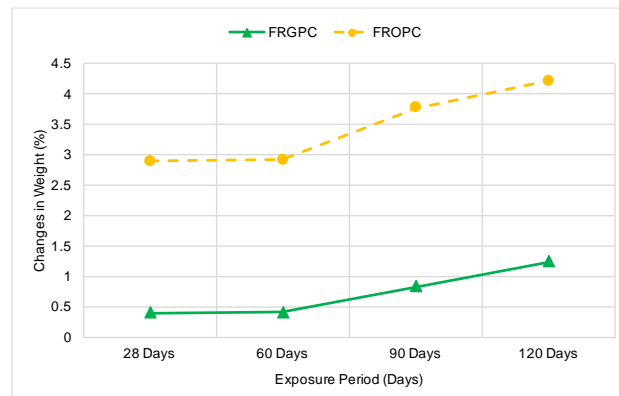


Figure 2. Changes in weight of FRGPC and FROPC.

The FRGPC performed a steady weight change at 28 days and 60 days and gradual increase for the following days. FRGPC has low weight change percentages compared to FROPC indicate that there is no sign of chloride accumulation in the samples [29]. A marginal microstructure alteration is occurred in the concrete sample as FRGPC concrete after 28 days showed a consistent weight change with time. In contrast, a gradual increase in the percentage of weight change with time is shown in FROPC concrete. This may be due to accumulation of chloride crystal in the concrete pores that was bound by the paste and this will increase the final weight of the concrete. There is less chloride accumulation in the FRGPC concrete compared to FROPC concrete [29]. Therefore, the change in weight of the FRGPC concrete is minor in the continuous immersion related to the increase of porosity. FRGPC concrete shows good resistance to acid and very less change in weight throughout the test. The percentage difference for changes in weight between FRGPC concrete and FROPC concrete is 70.62%.

3.3. Carbonation Depth

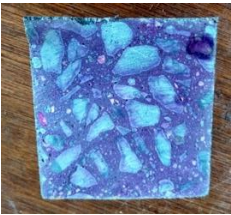

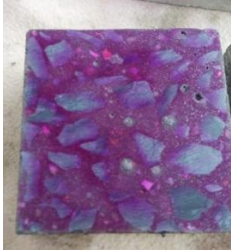



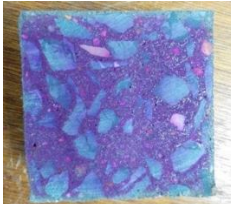

Table 3 depicts the change in colour on the freshly split surface of concrete between carbonated and non-carbonated region in both types of concrete which are FRGPC concrete and FROPC concrete using phenolphthalein indicator.

Based on the results obtained, there is no effect on carbonation for both FRGPC concrete and FROPC concrete. This is due to penetration depth is mostly found on outer layer of the samples when concrete is subjected to carbonation [30]. Dense microstructure of the sample concrete has slow rate of diffusion cause the carbonation is only occurred on the outer layer or surface of the concrete. Besides, immersion of concrete sample for 120 days might be too short for the carbonation process to occur. The strength of the concrete is affected by carbonation. Hence, the strength could be higher when the carbonation is not prominent. Furthermore, moisture content of concrete significantly affects the rate of carbonation [31]. The low moisture content of the concrete cause the carbon dioxide diffuses more rapidly. This is due to the diffusion if gas in air is rapid than in water. However, the diffusion of carbon dioxide is very low if the pore of concrete is fully filled with water when the concrete samples is fully immersed in seawater.

The carbonation of FRGPC concrete is different with FROPC concrete where there is no obvious boundary between the area of coloured region and colourless region compared to FROPC concrete. Change in colour on FROPC concrete is more obvious and rapid than geopolymer concrete after phenolphthalein indicator is sprayed on a freshly split concrete. Based on the research by Neville [32], occurrence of partial carbonation will cause unobvious carbonation 'front' on the freshly split concrete surface. The calcium hydroxide, Ca(OH)_2 and C-S-H gel are produced by FROPC concrete whereas FRGPC concrete produced $[\text{Mz}(\text{AlO}_2)_x(\text{SiO}_2)_y \cdot n\text{MOH} \cdot m\text{H}_2\text{O}]$ gel [33]. With the reason of Ca(OH)_2 and C-S-H are not present in FRGPC concrete, NaOH is the only constituent that can be carbonated.

Sodium carbonate, Na_2CO_3 is formed when NaOH reacts with CO_2 and releasing water [31]. The carbonation of NaOH is only partial carbonation while $\text{Ca}(\text{OH})_2$ or C-S-H is a full carbonation. The overall colour of non-carbonated FROPC concrete was much darker than in the non-carbonated FRGPC concrete and take shorter time for changes in colour after it was sprayed with phenolphthalein indicator.

Table 3. The change in color between carbonated and non-carbonated region in FRGPC and FROPC.

Days	FRGPC	FROPC
28		
60		
90		
120		

4. Conclusion

The trend for compressive strength of FRGPC is in decreasing trend as the time of seawater immersion increased. The highest compressive strength obtained by FRGPC concrete is 76.87MPa at 28 days whereas the lowest compressive strength obtained is 51.63MPa. This is due to porosity of concrete that cause ingress of sulfate ions into concrete in seawater. The longer the immersion time in seawater, the higher the amount of sulfate ions can be ingress into geopolymer concrete.

FRGPC concrete showed the increasing trend for changes in weight. This is due to the chloride penetration that has been bound into the paste to increase the density of concrete when immersed in seawater.

There is no effect for both FRGPC and FROPC concrete on carbonation progress. This is due to the time exposure to seawater is too short for the carbonation progress. The change in colour after phenolphthalein solution is sprayed is lighter or not clear and slower for GPC concrete when compared to OPC concrete with the reason of partial carbonation is occurred for GPC concrete.

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